Kootenai Development Company

Flyway Property

Quality Assurance Project Plan (QAPP)

April 2004 (draft revision 1)

Prepared by:

Remedium Group, Inc.
A Subsidiary of W. R. Grace & Co.
6401 Poplar Ave., Suite 301
Memphis, TN 38119-4840

DRAFT

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN

Kootenai Development Company

Flyway Property

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Kootenai Development Company

Flyway Property

Quality Assurance Project Plan (QAPP)

April 2004 (draft revision 1)

Prepared by: _____
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Reviewed by: _____
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Koch Environmental Health, Inc.

Reviewed by: _____
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Koch Environmental Health, Inc.

Reviewed by: _____
Mike Chapman
Mike Chapman Enterprises

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Analytical Laboratory

Approved by: _____
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EPA Region VIII Remedial Project Manager

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- Department of Public Health and Human Services Environmental Laboratory, State of Montana, Certification for Drinking Water Analysis, Expiration Date: 9/18/06

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Koch Environmental Health, Inc.
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Section 1
Project Management

This QAPP supports the draft Remedial Action Work Plan (RAWP) for the Flyway site located near Libby, Montana. This section covers the basic area of project management, including the project organization, background and purpose, project description, quality objectives and criteria, special training, and documentation and records.

1.1   Project Organization

Organization and responsibilities specific to this study are discussed in this section. Remedium Group, Inc. (Remedium) will provide the necessary technical and field staff to perform sampling and reporting aspects of the project. Health and Safety and Air Monitoring will be conducted by Koch Environmental Health, Inc. Excavation & Equipment will be supplied and conducted by Mike Chapman Enterprises. The analytical services will be provided through EMSL Analytical Laboratory (EMSL) located in Libby, Montana and Koch Environmental Health, Inc. (also Badlands Environmental Consultants, Inc.) located in Libby, Montana.

1.1.1 Remedium Management

The Remedium management team for the Flyway project is comprised of the following: Project Coordinator (PC), Alternate PC, Project Manager, Project Quality Assurance Coordinator, Sample Coordinator and Air Monitoring Manager, Excavation and Equipment Contractor, Health & Safety Officer, and project support team. See Figure 1-1 for the Project Management organization chart.

The following personnel are assigned to this project:

Project Coordinator   Robert Marriam

FIGURE 1-1
PROJECT MANAGEMENT ORGANIZATION CHART

```
┌─────────────────────┐        ┌─────────────────────┐
│  Remedium Group, Inc.│        │ Remedium Group, Inc. │
│  Alternate Project   │────────│ Project Coordinator  │
│  Coordinator         │        │ Robert Marriam       │
│  Robert Medler       │        │                      │
└─────────────────────┘        └─────────────────────┘
                                           │
                               ┌─────────────────────┐
                               │  W. R. Grace & Co.   │
                               │  Project Manager     │
                               │  Alan Stringer       │
                               └─────────────────────┘
                                           │
┌─────────────────────┐                    │
│Project Quality Assurance│                 │
│  Coordinator         │──────────────────┤
│  J. David Tucker     │                    │
└─────────────────────┘                    │
                               ┌─────────────────────┐
                               │ Koch Environmental   │
                               │ Health, Inc.         │
                               │ Sample Coordinator and│
                               │ Air Monitoring Manager│
                               │ Patrick McGurren     │
                               └─────────────────────┘
┌─────────────────────┐                    │
│  Data Validators     │──────────────────┤
│                      │                    │
└─────────────────────┘                    │
```

Koch Environmental Health, Inc. H & S Officer Thomas Koch, C.I.H.	Mike Chapman Enterprises Excavation & Equipment Mike Chapman, Operator	Field Team Leaders

Sample Personnel	Onsite GIS Personnel

Alternate PC	Robert J. Medler
Project Manager	Alan Stringer
Project Quality Assurance Coordinator	J. David Tucker
Sample Coordinator and Air Monitoring Manager	Patrick McGurren (Koch Environmental Health, Inc)
Excavation and Equipment Contractor	Mike Chapman (Mike Chapman Enterprises)
Health & Safety Officer	Thomas Koch, C.I.H. (Koch Environmental Health, Inc.)

The Remedium **PC and Alternate PC** for this removal action work will be Mr.

Robert Marriam and Mr. Robert J. Medler, respectively. Mr. Marriam and Mr. Medler will

be responsible for the overall management and coordination of the following activities:

- Maintaining communications with EPA regarding the status of this project;

- Supervising production and review of deliverables;

- Reviewing analytical results;

- Tracking of planned budgets and schedules;

- Incorporating and informing EPA of changes in the RAWP, HASP, and other project documents;

- Providing oversight of data management;

- Notifying the appropriate personnel immediately of significant problems affecting the quality of data or the ability to meet project objectives;

- Using sampling data in site remediation decision making;

- Preparing weekly status reports; and

- Reviewing analytical results.

The Remedium **Project Manager (PM)** for this removal action work is Mr. Alan

Stringer. Mr. Stringer is responsible for the daily management of the following activities:

- Overseeing excavation operations in the field;

- Notifying the PC of significant problems which may affect the quality of the data or the ability to meet project objectives;

- Scheduling personnel and material resources;

- Implementing sampling and analysis aspects of the cleanup;

- Organizing and conducting periodic meetings with onsite facility personnel;

- Providing oversight of daily and periodic report preparation;

- Coordinating work activities including sampling;

- Ensuring that sampling is conducted in accordance with pertinent procedures and that the quantity and location of all samples meet the requirements of this RAWP; and

- Scheduling and conducting required sampling and monitoring activities.

The **Project Quality Assurance Coordinator** for this project will be Mr. David Tucker. The Project Quality Assurance Coordinator has the authority to objectively review projects and identify problems, and the authority to use corporate resources as necessary to resolve any quality-related problems. The Project Quality Assurance Coordinator for this project will be responsible for the following:

- Verifying that corrective actions resulting from staff observations, QA/QC surveillances, and/or QA audits are documented and implemented;

- Reviewing and approving the project-specific plans;

- Directing the overall project QA/QC program;

- Maintaining QA/QC oversight of the project;

- Reviewing QA/QC sections in project reports, as applicable;

- Reviewing QA/QC procedures applicable to this project;

- Initiating, reviewing, and following up on response actions, as necessary;

- Take corrective action as needed;

- Arranging performance audits of measurement activities, as necessary; and

- Providing weekly written reports on the QA/QC activity to the QA manager.

The **Sample Coordinator and Air Monitoring Manager** for this project is Mr. Patrick McGurren. Mr. McGurren is responsible for the following:

- Maintaining proper chain-of-custody forms and sample labels for proper transfer of the samples to the analytical laboratories;

- Preparing and shipping samples to the analytical laboratories; and

- Maintaining sampling equipment.

Mr. Patrick McGurren will receive the soil sample analysis data directly from EMSL, and ambient and personal air sample analysis data from Koch Environmental Health, Inc. (also Badlands Environmental Consultants, Inc.) in the Libby 2 format. Remedium will also provide a QA/QC review of the field data package, ensuring that the data, with backup instrument calibration and standard information, is included.

Mr. Patrick McGurren will conduct the soil sampling and the air sampling as per indicated in the SAP.

The **Excavation and Equipment Contractor** for this excavation and sampling effort is Mr. Mike Chapman (Mike Chapman Enterprises). Mr. Chapman is responsible for the overall management and coordination of the following activities:

- Overseeing operation and maintenance of excavation and equipment activities; and

- Conducting tailgate safety meetings with truck drivers.

The **Health & Safety Officer** for this project is Mr. Thomas Koch, C.I.H. (Koch Environmental Health, Inc.). Mr. Koch is responsible for the overall health & safety of the workers and work efforts conducted as part of this project. See Appendix A for a copy of the Health & Safety Plan (HASP).

Section 2
Background and Purpose

Site background and history are provided in the RAWP and the SAP. The purpose and objectives of the sampling and analysis efforts are discussed in the RAWP and the SAP. The purpose of this QAPP is to provide guidance to ensure that all environmentally related data collection procedures and measurements are scientifically sound and of known, acceptable, and documented quality, and conducted in accordance with the requirements of the project.

2.1   Project Description

A description of this project is provided in the RAWP and SAP. Work efforts will only be conducted in the 53-Grid Area (see Figure 2-1 for the 53-Grid Area).

No work efforts will be conducted in the Riverbank area; this area has already been remediated.

No work efforts will be conducted in the Transformer area. No PCB soil contamination was found in this area, according to EPA's soil sample results.

2.2   Quality Objectives and Criteria for Measurement

This section provides an internal means for control and review of the project, so that environmentally related measurement and data collected are of known and acceptable quality. The data quality objectives (DQOs) and data measurement objectives are addressed below.

In support of the quality objectives and criteria for measurement, the following documents are attached to this QAPP.

Appendix 1

- Request for Modification to the Libby sampling and Quality Assurance Project Plan Field Activities LFO-000000

Legend

$\bigwedge$ Approx. Flyway Property Boundry

☐ 100' Grid Tile

☐ Grids Requiring Removal

100   0   100   200 Feet

FIGURE 2-1
FLYWAY PROPERTY
LIBBY, MONTANA
53 GRID AREA

Remedium Group Inc.

Data Source - Plan by CDM
Entitled " Libby, Montana Figure A-2-2 "

Plan Prepared By:
Hayes & Associates
Woburn, Massachusetts

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- Department of Public Health and Human Services Environmental Laboratory, State of Montana, Certification for Drinking Water Analysis, Expiration Date: 9/18/06

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Appendix 7

- Badlands Environmental Consultants, Inc., Quality Manual, 1/5/00

2.3   Data Quality Objectives

The Data Quality Objectives (DQO) process is a series of planning steps based on scientific methods that are designed to ensure the type, quantity, and quality of environmental data used in decision-making are appropriate for the intended purpose.

2.3.1 Organization

To accomplish the project goals, the SAP calls for the sampling and analysis of ambient air, personal air, and soil. For convenience and to clarify the specific purpose of each sampling and analysis program, the DQOs are organized separately by medium and respective purpose. Whenever possible, this is accomplished in tabular form. As shown, the various DQOs are segregated into the following: (1) Personal Air (BZ)

Monitoring Samples, (2) Perimeter Monitoring Air Samples, and (3) Soil Confirmation Samples.

All project personnel are detailed in Section 1.1. The decision makers for the activities described in this SAP are Robert Marriam (PC), Robert Meddler (alternate PC), Alan Stringer (PM), David Tucker (PQAC), Patrick McGurren (Sample Coordinator and Air Monitoring Manger), Thomas Koch (H&S Officer), and Jim Christiansen (EPA RPM).

Previous investigations at the Flyway near Libby (see CDM's Final Removal Action Work Plan dated August 14, 2001) and CDM's QAPP were designed to characterize Libby amphibole asbestos (LA) contamination at that location. Removal activities will be performed at the Flyway in the fifty-three (53) grid area that have been found to contain LA asbestos-contaminated soils. During removal activities, the potential for LA fibers to migrate offsite increases. Likewise, during these activities, the potential for LA exposure to workers is also increased. Therefore, it is important to ensure worker safety and contaminant containment through periodic monitoring. Following cleanup (soil removal), confirmation soil samples will be collected and analyzed expeditiously to determine if the removal actions met project goals (categories) as described in Remedium's SAP dated April 2004. Therefore, a program must be put in place to monitor: (1) worker exposure and contaminant containment during removal activities; and (2) the effectiveness of the cleanup (i.e., soil sample confirmation) following removal activities.

Other areas previous identified at the Flyway site that will not need to be addressed is the Riverbank area and the Transformer area located near an abandoned pump house. Based on available information provided from previous work efforts, the Riverbank contaminated soil has already been addressed; therefore, no sampling and/or remedial activities are proposed for the soil in this area. Based on EPA's composite soil

sample data, no PCB contamination was detected beneath the Transformer area (near the abandoned pump house); therefore, no sampling and/or remedial activities are proposed for the soil in this area.

2.3.2 Principal Study Questions and Identify the Decision

The necessary resources (e.g., personnel, equipment, etc.) and budget will be allocated to meet the data quality objectives. The schedule of the proposed sampling, analysis, and removal response activities is located in the RAWP.

The principle study question(s), alternative actions, and decision statements are summarized in Table 2-1.

Table 2-1
Identify the Decision

Data Quality Objective	Sample Description	Principle Study Question(s)	Alternative Actions	Decision Statements
RA Monitoring	Personal (BZ) Air Monitoring	Is LA asbestos detected in the workers' breathing zone above worker safety limits?	1) Continue contaminated soil removal and re-evaluate engineering controls, work practices, and/or PPE 2) Stop work 3) Take no action	Are LA asbestos fibers collecting in the workers' breathing zone above worker safety limits? If yes, engineering controls, work practices, and/or PPE will be re-evaluated and/or work will stop. If no, cleanup activities will continue with no additional evaluation.
RA Monitoring	Perimeter Air Monitoring	Are LA asbestos fibers detected in air along the perimeter boundary of an exterior cleanup site (exclusion zone)?	1) Continue contaminated soil removal and re-evaluate engineering controls and work practices 2) Stop Work 3) Take no action	Are LA fibers migrating to the exclusion zone boundary during LA contaminated soil removal? If yes, engineering controls and/or work practices will be re-evaluated and/or work will stop. If no, excavation activities will continue with no additional evaluation.
RA Confirmation	Soil Confirmation Sampling	Is LA asbestos detected in the soil surface of the excavated area, after soil removal? If so, has the maximum excavation depth of 18 inches or 4 feet (soil contamination only or ACM/mine waste contamination, respectively) been achieved? See the SAP for each soil removal and sampling category.	1) Remove additional soils by either excavation or surface scraping 2) Stop removal and designate as either a non-contaminated area or an area of no further removal action See the SAP for each soil removal and sampling category.	If LA is detected, and – Max. excavation depth is not achieved: 1) Remove additional soils 2) Continue until no LA asbestos is detected or max. excavation depth achieved 3) Stop removal and designate as a non-contaminated area (if [LA] is ND) Max. excavation depth is achieved: 1) Stop removal and designate as either a non-contaminated area (if [LA] is ND) or an area of no further removal action (if [LA]<1%) 2) Stop work at a depth of 4 feet bgs. See the SAP for each soil removal and sampling category.

RA - Removal Action
ACM - Asbestos Containing Material
BZ - Breathing Zone
PPE - Personal Protective Equipment
bgs - below ground surface

a - Up to a depth of 18 inches, we require a non-detect via NIOSH 9002 to state that excavation is complete. At a depth of 18 inches, which is our general maximum excavation depth, we will only continue excavation if there is substantial asbestos containing material (such as mine waste) or samples via NIOSH 9002 are greater than or equal to 1%. See the SAP for each soil removal and sampling category.

2.3.3 Inputs to the Decision

The information needed for the decision, the action levels, the basis for the action levels, and analytical method summaries are provided in Table 2-2. Further details about the sampling and analysis methods that can meet the data requirements are summarized in the SAP (Section(s) 3, 4, and 5). This SAP is designed only for cleanups for which LA characterization at the Flyway property (e.g., soil concentration) has been performed through another investigation (SAP). Analytical results (that are confirmatory and do not serve to characterize contamination) are needed within hours of sampling so that excavation/cleanup work may continue with relative continuity. As such, confirmation soil samples will not be ground as in previous characterization studies and will be analyzed via polarized light microscopy (PLM) Method NIOSH 9002. Further, analyses will generally be performed onsite at EMSL's Libby laboratory to ensure expedited results.

Table 2-2
Inputs to the Decision

Data Quality Objective	Sample Description	Information Needed	Action Level[h]	Basis for Action Level	Analytical Method
RA Monitoring	Personal (BZ) Air Monitoring	AS_{PCM}: 1 f/cc AS_{TEM}: 0.1 LA s/cc Min. Volume: 400 L[a] Collect: TWA: 8-hour TWA STEL: 30 minute excursion sample	TWA: 0.1 PCME s/cc STEL: 1.0 s/cc	OSHA Worker Safety Regulations	PCM: NIOSH 7400 TEM[d]: TEM AHERA
RA Monitoring	Perimeter Air Monitoring	AS_{TEM}: ~0.005 LA AHERA/cc Min. Volume: 1200 L Collect: 4 samples, min. along north, south, east & west boundaries of EZ	Each air sample <AS_{TEM} Approx. 0.005 AHERA s/cc	Removal Action Clearance Criteria[b]	TEM AHERA
RA Confirmation	Soil Confirmation Samples	Reported Result: % LA by VAE AS: Method defined as 1%, but qualitative estimates of LA present below 1% reported as trace or ND Approx. Mass: 1 kg[e]	Up to max. cleanup depth of 18 inches: ND Beyond max. cleanup depth: ≥1% LA by VAE[b,c]	Removal Action Clearance Criteria[b,c]	Preparation: Homogenize, Cone & Cut Analysis: NIOSH 9002

AS-Analytical Sensitivity
RA - Removal Action
ND - Not Detected or non-detect
VAE - Visual Area Estimation
f/cc - Fiber Per Cubic Centimeter
bgs - Below Ground Surface
PCM - Phase Contrast Microscopy
TWA - Time Weighted Average
STEL - Short-term Exposure Limit
AHERA s/cc - AHERA structures per cubic centimeter of air
TEM AHERA - All samples are analyzed by transmission electron microscopy using the counting method as described in the Asbestos Hazard Emergency Response Act (AHERA) (EPA 1987) with project specific modifications.
EZ - Exclusion Zone

a - Minimum volume requirements according to the method are 25 L. However, in order to achieve a reasonable analytical sensitivity by TEM, the sampler should attempt to collect 400 L of air for the BZ sample.

b - Action Level/Clearance Criteria Technical Memorandum (EPA 2003).

c - In the spirit of statements made in the technical memorandum (EPA 2003), efforts will be made to avoid having to repeat cleanup activities by cleaning soils to ND up to the maximum cleanup depth of 18 inches. Excavation beyond the maximum cleanup depth will only continue if soils have concentrations ≥1% LA. Excavation will stop at a maximum depth of 4 feet bgs.

d - If PCM results are above the OSHA limit, TEM confirmation must be performed.

e - Approximately 0.5 kg for analysis and 0.5 kg for EPA split sample (when required).

2.3.4 Define the Study Boundaries

The target population, spatial and temporal boundaries, potential constraints, and the smallest subpopulation are summarized in Table 2-3.

Table 2-3
Study Boundaries

Data Quality Objective	Sample Description	Target Population	Spatial Boundaries	Temporal Boundaries[b]	Potential Constraints	Smallest Sub-population
RA Monitoring	Personal (BZ) Air Monitoring	Ambient air within the workers' breathing zone; during removal activities	Each individual worker's breathing zone for the activity type performed	Collected during exterior removal activities (ie., excavation)	NA	1) Air sample for each work activity per week. DEFINE THE WORK ACTIVITIES HERE
RA Monitoring	Perimeter Air Monitoring	Ambient air at the boundary of the EZ; during removal activities	Vertical: Air space above the exclusion zone to sampling height (~4-6 ft.) Horizontal: perimeter bounding the site-specific EZ	Collected during exterior removal activities (ie., excavation)	Inaccessibility due to property boundaries or other obstacles Inclement weather such as rain that can cause the sample to be void[c]	1) 4 air samples that bound the EZ
RA Confirmation	Soil Confirmation Sampling	Surface soil at the bottom of the excavation site; after soil removal activities	Vertical[a]: Generally: 18 inches bgs to ground surface Maximum: 4 feet bgs to ground surface Horizontal: site-specific grid area	Collected after all contaminated soil is excavated and removed from the site and will continue until the area is designated as either non-contaminated or removal actions are discontinued (no further action)	No soil available for sampling because excavation continued to bedrock	1) A composite soil sample for every 100 ft² excavated See the SAP for each soil removal and sampling category

BZ – Breathing Zone
EZ - Exclusion Zone
RA - Removal Action
bgs - below ground surface
NA - Not Applicable

a - These are generally the vertical boundaries for soil. If LA contamination 31% is found, the vertical boundary shall be extended for that location until the concentration is below 1% LA or until a depth of 4 feet bgs is achieved (whichever is achieved first).
b - A general schedule/timeline for cleanups is provided in the Flyway RAWP. This section is specific to timeframes for sampling at the 53-grid area.
c - If it is raining, attempts will be made to protect the sample from moisture.

2.3.5 Decision Rule

The population parameter, action levels, and decision rules are summarized in Table 2-4.

Table 2-4
Decision Rule

Data Quality Objective	Sample Description	Population Parameter	Action Level	Decision Rule
RA Monitoring	Personal (BZ) Air Monitoring	1) Air sample representing the breathing zone for the activity conducted	TWA: 0.1 PCME s/cm^3 STEL: 1.0 s/cm^3	If the concentrations of the BZ samples ∃0.1 s/cm^3 (TWA) or ∃1.0 s/cm^3 (STEL) engineering controls, work practices, and/or PPE will be re-evaluated and/or work will stop. If concentrations are not above action levels, cleanup activities will continue with no additional evaluation.
RA Monitoring	Perimeter Air Monitoring	1) 4 air monitoring samples that bound the perimeter of the EZ	Each air sample <AS$_{TEM}$ Approx. 0.005 AHERA s/cm^3	If the concentration of any of the 4 samples ∃0.005 AHERA s/cm^3, then excavation engineering controls and work practices will be re-evaluated and/or work will be stopped. If all 4 perimeter air samples are ND, then no action will be taken.
RA Confirmation	Soil Confirmation Samples	1) Composite soil sample representing the area of excavation, per 100 ft^2	Up to max. cleanup depth of 18 inches: ND Below max. cleanup depth (& up to 4 feet bgs): <1% LA by VAE^{a,b}	If LA is detected, and – Max. excavation depth is not achieved: 1) Remove additional soils 2) Continue until no LA asbestos is detected or max. excavation depth achieved 3) Stop removal and designate as a non-contaminated area (if [LA] is ND) Max. excavation depth is achieved: 1) Continue removing additional soils IF [LA] ∃1% 2) Stop removal and designate as either a non-contaminated area (if [LA] is ND) or an area of no further removal action (if [LA]<1%) 3) Stop work at a depth of 4 feet bgs.

BZ – Breathing Zone
EZ - Exclusion Zone
RA - Removal Action
ND - Not Detected or Non-detect
PCME - Phase Contrast Microscopy Equivalent
TWA - Time Weighted Average
STEL - Short-term Exposure Limit

a - Action Level/Clearance Criteria Technical Memorandum (EPA 2003).
b - In the spirit of statements made in the technical memorandum (EPA 2003), efforts will be made to avoid having to repeat cleanup activities by cleaning soils to ND up to the maximum cleanup depth of 18 inches. Excavation beyond the maximum cleanup depth will only continue if soils have concentrations ∃1% LA. Excavation will stop at a maximum depth of 4 feet bgs.

2.3.6 Tolerable Limits on Decision Errors

For the purposes of completing all six steps of the DQO process, the null hypotheses, consequences of making an incorrect decision, gray region, and tolerable limits are summarized in Table 2-5.

This DQO process is useful to encourage careful design of decision rules by defining and integrating the errors that are acceptable based upon a myriad of integrated project management decisions such as reduction in risk to human health, implementability/practability, and cost. As stated in the guidance document for development of DQOs: QA/G-4 (EPA 2000), solely statistically generated tolerable limits on decisions errors, are not necessary in certain cases, providing a line of reasoning (scientific justification) is presented that adequately defines acceptable limits or decision errors. This particular effort was put forth in the Action Level/Clearance Criteria Technical Memorandum (EPA 2003) for the following DQOs: (1) Asbestos in Soil Confirmation Samples and (2) Perimeter Monitoring Air Samples. The decision rule for the personal (BZ) air monitoring samples has been promulgated by legislation, and as such, limits on decision errors do not apply.

Table 2-5
Limits on Decision Errors

Data Quality Objective	Sample Description	Null Hypothesis	Type I Error — Will Result in:	Type II Error — Will Result in:	Gray Region	Tolerable Limits
RA Monitoring	Personal (BZ) Air Monitoring	1) The BZ air is contaminated with LA above the worker safety action levels.	1) Determining that the BZ air is not contaminated with LA above the worker safety action levels when it actually is. This in turn, results in and increased risk to workers performing removal actions.	1) Determining that the BZ air is contaminated with LA above the worker safety action levels when it is not. This in turn, results in re-evaluating engineering controls, possibly stopping work, or increasing the level of PPE when it is not necessary and adds unnecessarily to cleanup costs.	NA	NA
RA Monitoring	Perimeter Air Monitoring	1) The perimeter air is contaminated with LA.	1) Determining that the perimeter air is not contaminated with LA when it actually is. This in turn, results in an increased risk to human health.	1) Determining that the perimeter air is contaminated with LA when it is not. This in turn, results in re-evaluating engineering controls and possibly stopping work when it is not necessary, and adds unnecessarily to cleanup costs.	NA	NA
RA Confirmation	Soil Confirmation Sampling	1) The soils below an excavation are still contaminated with LA after removal.	1) Determining that the surface soils at the bottom of the excavated area are not contaminated with LA when they actually are. This in turn, results in an increased risk to human health.	1) Determining that the surface soils at the bottom of the excavated area are contaminated with LA when they are not. This in turn, results in excavation of additional soils when it was not necessary and adds unnecessarily to cleanup costs.	NA	NA

BZ – Breathing Zone
NA - Not Applicable
RA - Removal Action
PPE - Personal Protective Equipment

2.3.7 Optimize the Design for Obtaining Data

Using data previously generated for the site, the DQOs have been designed to support the proposed removal activities for the Flyway RAWP, and represents the best possible project planning effort. However, in implementing the SAP, unforeseen situations may arise, or team members may find more efficient means to carry out some of the day-to-day activities. Therefore, team members are always afforded the opportunity to recommend optimization the data gathering design. Recommendations must come through proper channels and documented using either a modification form or and addendum to the RAWP. All modifications or addendums must be approved prior to making the proposed changes.

Section 3
Laboratory Analysis

Soil samples collected under this QAPP will be analyzed for asbestos using the PLM analytical method (NIOSH 9002 Method). The ambient and personal air samples will be analyzed for PCM (NIOSH Method 7400) and a percentage will be analyzed using the TEM analytical method (AHERA TEM).

The soil samples will be submitted to EMSL's Libby laboratory for analysis.

The ambient and personal air samples will be submitted to Koch Environmental Health, Inc.'s (also Badlands Environmental Consultants, Inc.) Libby laboratory for analysis.

Prior to shipping samples, sampling personnel will ensure that the laboratory is ready to receive and analyze the samples, and can provide an electronic copy of the data in the Libby 2 format. In addition, the laboratories will submit analytical data reports to Remedium. The data reports will contain a case narrative that briefly describes the number of samples, the analyses, and any noteworthy analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include signed chain-of-custody forms, container receipt forms, analytical data, and a QC package. The laboratories will also provide an electronic copy of the data to Remedium.

3.1 Reporting Limits

The reporting limits are the minimum levels that the laboratories will report without a qualifier. It is therefore important for the laboratory to monitor the sensitivity of data-gathering instruments to ensure data quality through constant instrument performance checks.

3.2 Holding Times and Preservation

There are no required holding times or parameters for asbestos.

3.3 Quality Control Analyses

Project analytical laboratories will follow all laboratory QC requirements as outlined in their respective statements of work, or in the handbook of laboratory analytical methods and references (EPA 2003), as applicable. Laboratory QC may be measured by the preparation and analysis of laboratory duplicates, MSs, LCSs, and/or laboratory blanks (i.e., preparation blanks), or by visual verification or other controls consistent with national standardized laboratory operation programs (e.g., National Voluntary Laboratory Accreditation Program criteria).

3.4 Special Training Requirements

Special training required for this study may include the following:

- Health and safety training, as described in the HASP, including 40 hour Occupational Safety and Health Administration (OSHA) Training and 8 Hour Refresher Training; and

- Asbestos Inspector Training.

3.5 Documentation and Records

The laboratories will submit the sample data packages in a hard copy and in an electronic version (pf) to the Remedium laboratory coordinator, as required by the Remedium subcontract with the laboratory. An electronic data deliverable (EDD), in the Libby 2 format, will also be provided to Remedium's Project Quality Assurance Coordinator.

Section 4
Measurement and Data Acquisition

This section covers sample process design, sampling methods requirements, handling and custody, analytical methods, QC, equipment maintenance, supply acceptance, and data management. The field procedures are designed so that the following occurs:

- Samples collected are consistent with project objectives; and
- Samples are collected in a manner so that data represent actual conditions.

4.1   Sample Process Design

The general goal of sampling is to provide information regarding soil confirmation and perimeter air monitoring, during and after soil excavation at the Libby site.

4.2   Sampling Methods Requirements

Sampling methods, sample containers, and overall field management is described below.

4.2.1 Sampling Equipment and Preparation

Equipment required for field sampling is listed in the SAP and in Appendix 8.

4.2.2 Sample Containers

Sample containers required for field sampling will consist of a 1-gallon zip lock plastic bag for the soil samples and air sample cassettes for the air samples.

4.2.3 Sample Collection, Handling, and Shipment

Samples collected during the study consist of soil, air, and QC samples. All samples will be handled and shipped according to procedures located in Appendix 8.

4.3 Sample Handling and Custody Requirements

Custody and documentation for field and laboratory work are described below, followed by a discussion of corrections to documentation.

4.3.1 Sample Handling and Field Custody Procedures

This section describes sample labeling, field custody procedures, and sample handling.

4.3.1.1   Sample Labeling and Identification

A unique alphanumeric code will identify each sample collected during sampling events. The coding system will provide a tracking record to allow retrieval of information about a particular sample and to ensure that each sample is uniquely identified. Sample numbers will correlate with locations to be sampled. The sample locations and numbers will be identified in the field logbooks.

Samples will be labeled with index identification numbers supplied and maintained by the sample coordinator, and signed out by the sampling teams.

Labels will be used in accordance with the Sample Custody Form. Sample labels will be completed and affixed to the appropriate sample containers. Preprinted adhesive labels may be used. These labels will be secured with waterproof tape if necessary.

4.3.1.2   Chain-of-Custody Requirements

Chain-of-custody procedures and sample shipment will follow the requirements stated in the procedure located in Appendix 8. The chain-of-custody record is employed as physical evidence of sample custody and control. This record system provides the means to identify, rack, and monitor each individual sample from the point of collection through final data reporting. A completed chain-of-custody record is required to

accompany each shipment of samples. A sample chain-of-custody form from EPA is located in Appendix 1.

4.3.1.3   Sample Packaging and Shipping

Samples will be packaged and shipped in accordance with the packaging and shipping procedure (See Appendix 8) and laboratory. These samples will be placed in a plastic bag and in a container.

Custody seals will be placed over at least two sides of the container and secured by tape, if custody is released to a non-sampler. All samples will be delivered to the laboratory.

4.3.1.4   Field Logbook and Records

Field logbooks will be maintained in accordance with the field logbook procedure, located in Appendix 8. The log is an accounting of activities at the site, and will duly note problems or deviations from the governing plans and observations relating to the sampling and analysis program. The Flyway Project Coordinator will maintain the logbook(s).

4.3.2 Laboratory Custody Procedures and Documentation

Laboratory custody procedures are provided by the laboratory. Upon receipt at the onsite laboratory, each sample shipment will be inspected to assess the condition of the shipping container and the individual samples. The enclosed chain-of-custody records will be cross-referenced with all of the samples in the shipment. These records will be signed by the laboratory sample custodian, and copies will be provided to Remedium in the laboratory report. The sample custodian will continue the chain-of-custody record process by using the chain-of-custody record number for each sample on

receipt. It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, data reporting, and disposal.

4.3.3 Corrections to and Deviations from Documentation

Documentation modification requirement for field logbook entries are described in the field logbook procedure (see Figure 8). For the logbooks, a single strikeout, initialed and dated, is required for documentation changes. The correct information should be entered in close proximity to the erroneous entry.

All deviations from the guidance document controls will be recorded in field logbooks. In addition, any major deviations to field sampling procedures will be documented on a Record of Deviation/Request for Modification Form, which will undergo review by Remedium prior to implementation of field changes. Any modifications to chain-of-custody forms will be made on the sample coordinator copy of the form and faxed to the analytical laboratory for documentation purposes.

4.4 Analytical Methods Requirements

The laboratory QA program and analytical methods are addressed below.

4.4.1 Laboratory Quality Assurance Program

Samples collected during this project will be analyzed in accordance with the specific EPA procedure. The purpose of using the specific procedure is to provide analytical data of known quality and consistency. Analytical laboratories will adhere to QC requirements as established the analytical method used.

4.4.2 Methods

The methods to be used for asbestos analysis are presented in Section 3. In addition, the Appendices include the laboratory SOPs.

4.5 Quality Control Requirements

Field, laboratory, and internal office QC are discussed below.

4.5.1 Field Quality Control Samples

4.5.1.1 Ambient Air

Field QC samples will be collected. See Section 3.3.6 of the SAP for the ambient air QC samples.

4.5.1.2 Personal Air

Field QC samples will be collected. See Section 4.3.6 of the SAP for the personal air QC samples.

4.5.1.3 Soil

Field QC samples will not be collected. See Section 5.3.4 of the SAP for the soil QC samples.

4.5.2 Laboratory Quality Control

Project analytical laboratories will follow all laboratory QC requirements as outlined in their respective statements of work, or in the handbook of laboratory analytical methods and references (EPA 2003), as applicable. Laboratory QC may be measured by the preparation and analysis of laboratory duplicates, MSs, LCSs, and/or laboratory blanks (i.e., preparation blanks), or by visual verification or other controls consistent with national standardized laboratory operation programs (e.g., National Voluntary Laboratory Accreditation Program criteria). See the appendices for the laboratory certifications and SOPs.

4.5.2.1 Laboratory Internal Quality Control Samples

QC data are necessary to determine precision and accuracy, and to demonstrate the absence of interferences and/or contamination. Each type of laboratory-based QC sample will be analyzed in accordance with the laboratories SOPs. The results of the QC analysis will be included in the QC package, and QC samples may consist of laboratory duplicates, and laboratory blanks, whichever is applicable, and any other method-required QC samples.

4.5.2.2 Laboratory Quality Control Checks

The laboratories will perform the QC checks required by each analytical method.

4.5.3 Internal Quality Control Checks

Internal QC checks will be conducted throughout the project to evaluate the performance of the project team during data generation. All internal QC will be conducted in accordance with the applicable procedures listed below:

- All project deliverables will receive technical and QA reviews prior to being issued to EPA in any form;

- Completed review forms will be maintained in the project files; and

- Corrective action of any deficiencies is the responsibility of the PM.

4.6 Equipment Maintenance Procedures

All field and laboratory equipment will be maintained in accordance with the manufacturers' maintenance and operating procedures.

4.7 Instrument Calibration Procedures and Frequency

Calibration of field and laboratory instruments is addressed in the following subsections.

4.7.1 Field Instruments

The only field instruments utilized will be the pumps to collect the air samples.

4.7.2 Laboratory Instruments

Calibration of laboratory instruments will be based on written procedures approved by laboratory management and included in the laboratory's QA manual. Instruments and equipment will be initially calibrated, and will continuously be calibrated at required intervals as specified by either the manufacturer or by more updated requirements (e.g., methodology requirements). Calibration standards used as reference standards will be traceable to EPA, National Institute of Standards and Technology, or another nationally recognized reference standard source.

Records of initial calibration, continuing calibration, repair, and/or replacement of laboratory equipment will be filed and maintained by the laboratories. Calibration records will be filed and maintained at the laboratories' location, where the work is performed, and may be required to be included in data reporting packages.

4.8 Acceptance Requirements for Supplies

Prior to acceptance, all supplies and consumables will be inspected by the Project Manager and/or team leaders to ensure that the supplies and consumables are in satisfactory condition and free of defects.

4.9 Nondirect Measurement Data Acquisition Requirements

Nondirect measurement data includes information from previous sampling events. The acceptance criteria for such data include a review by someone other than the author. Any measurement data included in information from the above sources (i.e., previous sampling event) will determine further action at the site, only to the extent that the data can be verified by project staff.

4.10 Data Management

Analytical results are maintained in the Libby version 2 secured project database.
Hard copy data reports will be maintained in the project files in the Remedium office in
Libby, Montana.

Section 5
Assessment and Oversight

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required, and that deviations from procedures are documented. These reports also serve to keep management current on field activities.

5.1   Assessments and Response Actions

Assessments and corresponding response actions are discussed below. It is important to note that EPA may perform onsite field and/or laboratory analysis or visits at any time.

5.1.1 Assessments

Performance assessments are quantitative checks on the quality of a measurement system and are appropriate to analytical work. Performance assessments for the laboratories may be accomplished by submitting reference material as blind reference (or performance evaluation) samples. These assessment samples are samples with known concentrations, that are submitted to the laboratories without informing the laboratories of the known concentration or that they are performance samples. Samples will be provided to the laboratories for performance assessment upon request from the EPA RPM. Laboratory audits may also be conducted upon request from the EPA RPM.

System assessments are qualitative reviews of different aspects of project work, to check on the use of appropriate QC measures and the functioning of the QA system. Any determination or change for project assessments will be performed by the Remedium Project Coordinator. Due to the amount of sampling and the duration of the project, both a field audit and an office audit are scheduled for the site annually.

5.1.2 Response Actions

Response actions will be implemented on a case-by-case basis to correct quality problems. Minor response actions taken in the field to immediately correct a quality problem will be documented in the applicable field logbook, and a verbal report will be provided to the Remedium PC. For verbal reports, the Remedium PC will complete a communication log to document that response actions were relayed to him. Major response actions taken in the field will be approved by the Remedium PC and the EPA RPM prior to implementation of the change. Major response actions are those that may affect the quality or objective of the investigation. Quality problems that cannot be corrected quickly through routine procedures may need to be documented in writing.

5.2   Reports to Management

QA reports will be provided to management whenever quality problems are encountered. Field staff will note any quality problems on field data sheets, or in field logbooks. Remedium's PC will be informed immediately and will be corrected. Weekly reports and change request forms are not required for this work assignment. Monthly QA reports will be developed by the project PC.

- Topics to be summarized regularly may include but not be limited to:
- Document technical and QA reviews that have been conducted;
- Activities and general program status;
- Project meetings;
- Corrective action activities;
- Any unresolved problem; and
- Any significant QA/QC problems not included above.

The EPA will receive copies of all management reports.

Section 6
Data Validation and Usability

Laboratory results will be reviewed for compliance with project objectives. Data validation and evaluation are discussed in the laboratory SOPs located in the appendices.

6.1   Data Review, Validation, and Verification Requirements

Due to the real-time usage of air monitoring and confirmation soil sampling results, no formal data validation for these media is currently required of Remedium. However, data is reviewed daily by the field health and safety coordinator and the project manager to ensure data (e.g., sampling dates and sample volumes, as appropriate) are reported correctly by the analytical laboratory. In addition, data packages are reviewed for completeness prior to distribution.

The analytical laboratories will validate the soil and air data. Data validation consists of examining the sample data package(s) against pre-determined standardized requirements. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, chain-of-custody information, raw data, LCS/LCSDs, MS/MSDs, initial and continuing instrument calibration, and other reported information to determine the accuracy and completeness of the data package. During this process, the validator will verify that the analytical methodologies were followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information. Analytical results will then be qualified as necessary.

Data verification includes checking that results have been transferred correctly from laboratory data printouts to the laboratory report and to the Libby 2 electronic data deliverable (EDD). Data verification for this project is primarily performed as a function

of built-in quality control checks in the Libby project database when data is uploaded. However, Remedium's project quality assurance coordinator will notify the laboratories and Remedium's PC of any discrepancies found during data usage.

6.2   Reconciliation with Data Quality Objectives

Once data has been generated, the analytical laboratories will evaluate the data to determine if DQOs were achieved. This achievement will be discussed in the measurement report, including the data and any deviations to the RAWP. Sample data will be maintained in a Libby 2 database. Laboratory QC sample data will be stored in hard copy (in the project files) and in a separate database, to be determined.

Appendix 1

- Request for Modification to the Libby Sampling and Quality Assurance Project Plan Field Activities LFO-000000

- Libby Asbestos Investigation – Chain of Custody Record

- Libby Field Sample Data Sheet (FSDS) For Personal Air

- Libby Field sample Data Sheet (FSDS) For Soil

- Libby Field Sample Data Sheet (FSDS) For Stationary Air

Request for Modification
to the
Libby Sampling and Quality Assurance Project Plan
Field Activities
LFO-000000

Project QAPP (circle one): Phase I (approved 4/00)   Phase II (approved 2/01)

Removal Action (approved 7/00)   Contaminant Screening Study (approved 5/02)

Other (Title and approval date): _____

SOP (Number and Revision No.):_____

Other Document (Title, Number/Revision):_____

Requester:_____   Title:_____
Company:_____   Date: _____

Description of Modification:
_____
_____
_____
_____

Field logbook and page number modification is documented on:_____

Reason for modification:
_____
_____
_____
_____

Duration of Modification (circle one):
  Temporary   Date(s):_____
                 Resident address(es): _____
                 _____

         -    If appropriate, attach a list of all applicable Index Identification numbers.

         Permanent (complete Proposed Modification Section) Effective Date:_____

Proposed Modification to SQAPP (attach additional sheets if necessary; state section and page
numbers of SQAPP when applicable):_____
_____

Technical Review and Approval: _____   Date:_____
(Volpe Project Manager or designate)

EPA Review and Approval: _____   Date:_____
(USEPA RPM or designate)

Page 1 of 1

Chain of Custody Record

From: _____

Libby Asbestos Investigation

Send to: _____

No. F0000

via: hand delivery   shipped

Sample Placed in Cooler/Bag	Index ID	Suffix ID	Sample Date	Sample Media (S=Soil; W=Water; D=Dust; A=Air; B=Bulk Insulation)	Volume (L) or Area (cm²)	Filter Pore Size (0.8μm or.45μm)	Turn Around Time	Analysis Request*	Comments	Sample Received by Lab

Stationary AIR: PCM (by NIOSH 7400 (Issue 2))   TEM-ISO 10312 (by ISO 10312:1995(E))   TEM-AHERA (AHERA).
Personal AIR: PCM (by NIOSH 7400 (Issue 2))   TEM-ISO 10312 (by ISO 10312:1995(E))   TEM-AHERA (AHERA).
SOIL: PLM-9002 (NIOSH 9002 (Issue 2))

Total Number of Samples _____   END OF SUBMITTAL

Additional Comments:_____

Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt

Chain of Custody Record

From: _____

_____

_____

Libby Asbestos Investigation

Send to: _____

_____

_____

No.   F0000

via: hand delivery   shipped

Stationary AIR: PCM (by NIOSH 7400 (Issue 2))   TEM-ISO 10312 (by ISO 10312:1995(E))   TEM-AHERA (AHERA).
Personal AIR: PCM (by NIOSH 7400 (Issue 2))   TEM-ISO 10312 (by ISO 10312:1995(E))   TEM-AHERA (AHERA).
SOIL: PLM-9002 (NIOSH 9002 (Issue 2))

Total Number of Samples _____   END OF SUBMITTAL

Additional Comments: _____

_____

Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt
Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt
Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt

March 31, 2004   Copies: Pink - Retained by Sample Coordinator; Yellow - Retained by laboratory; White - Included in analytical report   Page ___ of ___

LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR PERSONAL AIR

Field Logbook No: _____ Page No: _____ Sampling Date: _____

Address: _____ Owner/Tenant: _____

Business Name: _____

Land Use: Commercial   Mining   Roadway   Other (_____)

Sampling Team: REMEDIUM Other _____ Names:_____

Person Sampled: _____ SNN:_____ Task:_____

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS FB-(field blank) LB-(lot blank)	FS FB-(field blank) LB-(lot blank)	FS FB-(field blank) LB-(lot blank)
Matrix Type (circle)	Outdoor	Outdoor	Outdoor
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm
Pore Size (circle)	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8
Flow Meter Type (circle)	Rotometer DryCal NA	Rotometer DryCal NA	Rotometer DryCal NA
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No Yes NA	No Yes NA	No Yes NA
MET Station onsite?	No Yes NA	No Yes NA	No Yes NA
Sample Type	TWA EXC NA	TWA EXC NA	TWA EXC NA
Field Comments			
Cassette Lot Number: _____	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No
QC (Field Team) _____ Entered (LFO) _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____

For Field Team Completion (Provide Initials)	Completed by	QC by

vs 101503

LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR SOIL

Field Logbook No: _____   Page No: _____   Sampling Date: _____

⬤ Address: _____   Owner/Tenant: _____

Business Name: _____

Land Use: Commercial   Mining   Roadway   Other (_____)

Sampling Team: REMEDIUM   Other _____   Names:_____

Data Item	Sample 1	Sample 2	Sample 3
Index ID			
Location ID			
Sample Group			
Location Description (circle)	Grid _____ Other _____	Grid _____ Other _____	Grid _____ Other _____
Category (circle)	FS FD of _____ Field Blank (lot or equipment)	FS FD of _____ Field Blank (lot or equipment)	FS FD of _____ Field Blank (lot or equipment)
Matrix Type (Surface soil unless other wise noted)	Surface Soil Other _____	Surface Soil Other _____	Surface Soil Other _____
Type (circle)	Grab Comp. # subsamples _____	Grab Comp. # subsamples _____	Grab Comp. # subsamples _____
Sample Time			
Top Depth (in.)			
Bottom Depth (in.)			
Field Comments Note if vermiculite is visible in sampled area	BD-_____	BD-_____	BD-_____
Entered (LFO) _____	Remedium: Entered _____ Validated _____	Remedium: Entered _____ Validated _____	Remedium: Entered _____ Validated _____

For Field Team Completion (Provide Initials)	Completed by	QC by

LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR STATIONARY AIR

Field Logbook No: _____ Page No: _____ Sampling Date: _____

Address: _____ Owner/Tenant: _____

Business Name: _____

Land Use: Commercial   Mining   Roadway   Other (   )

Sampling Team: REMEDIUM Other _____ Names:_____

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS FB-(field blank) LB-(lot blank)	FS FB-(field blank) LB-(lot blank)	FS FB-(field blank) LB-(lot blank)
Matrix Type (circle)	Indoor Outdoor NA	Indoor Outdoor NA	Indoor Outdoor NA
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm
Pore Size (circle)	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8	TEM- .45 PCM- 0.8
Flow Meter Type (circle)	Rotometer DryCal NA	Rotometer DryCal NA	Rotometer DryCal NA
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No Yes NA	No Yes NA	No Yes NA
MET Station onsite?	No Yes NA	No Yes NA	No Yes NA
Sample Type	Pre Post Clear 2nd Clear 3rd Clear NA	Pre Post Clear 2nd Clear 3rd Clear NA	Pre Post Clear 2nd Clear 3rd Clear NA
Field Comments Cassette Lot Number: _____	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No	Archive Blank (circle): Yes No
QC (Field Team) _____ Entered (LFO) _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____	Volpe: Entered _____ Validated _____

For Field Team Completion (Provide Initials)	Completed by	QC by

vs 101503

Appendix 2

- EMSL Analytical, Inc. – Request for Modification to Laboratory Activities LB-000031, dated 2/5/04

- Syracuse Research Corporation – Request for Modification to Laboratory Activities LB-000030, dated 8/14/03

- Request for Modification to Laboratory Activities LB-000029a, dated 11/18/03

- Request for Modification to Laboratory Activities LB-000029, dated 8/26/03

- Request for Modification to Laboratory Activities LB-000028, dated 6/24/03

- Request for Modification to Laboratory Activities LB-000017A, dated 8/25/03

Request for Modification
To
Laboratory Activities
LB-000031

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other:_____

Requester: <u>R.K. Mahoney</u>   Title: <u>Senior Analyst / Special Projects Coordinator</u>

Company: <u>EMSL Analytical, Inc.</u>   Date: <u>27 January 2004</u>

Description of Modification:
<u>This is a clarification and expansion of TEM structure measurement and counting as expressed in the AHERA 40 CFR-Part 763 and ASTM D5755-95 methodologies.</u>

Reason for Modification:
<u>This clarification is intended to provide a basis for more consistent and uniform TEM results for the laboratories involved in the EPA Region 8, Libby, MT project.</u>

Potential Implications of this Modification:
<u>There are no negative potential implications of this clarification.</u>

Laboratory Applicability (circle one): ☒ All   Individual(s) _____

Duration of Modification (circle one):

    Temporary   Date(s): _____

                Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

    Permanent   (Complete Proposed Modification Section)   Effective Date: _____Historic_____

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

_____

_____

Technical Review: _____   Date: 27 January 2004
        (Laboratory Manager or designate)

Project Review and Approval: _____   Date: 1-27-04
        (Volpe: Project Technical Lead or designate)

Approved By:_____   Date: 2/5/04
    (USEPA: Project Chemist or designate)

A Guide for Structure Measurement and Classification.
AHERA 40 CFR – Part 763 and ASTM D5755-95
US EPA Region 8, Libby, MT Project
Laboratory Modification LB-000031

Figure 1   Simple fiber – Record length and width. Structure must meet AHERA length and aspect ratio criteria.

Figure 2   Stepped fiber – Record length. Record width as a best estimate of the average width. Structure must meet AHERA length and aspect ratio criteria.

Figure 3   Bundle – Record length and width. The aspect ratio of the overall structure is not a factor. At least three individual sub-structures in parallel arrangement separated by less than one sub-structure diameter, adequate to meet AHERA bundle definition, must meet AHERA length and aspect ratio criteria.

Figure 4   Stepped bundle – Record length. Record width as a best estimate of the average width. The aspect ratio of the overall structure is not a factor. At least three individual sub-structures in parallel arrangement separated by less than one sub-structure diameter, adequate to meet AHERA bundle definition, must meet AHERA length and aspect ratio criteria.

Figure 5   Matrix – Record longest exposed structure and its width. Structure must meet AHERA length and aspect ratio criteria.

Figure 6   Fiber with adhering matrix material - This structure does not fall into the matrix category as defined in that both ends are exposed (definition 14, AHERA) - Record length and width. Structure must meet AHERA length and aspect ratio criteria.

Figure 7   Structure with protrusions < 5:1 aspect ratio but an overall > 5:1 aspect ratio- Provided that the structure can be observed to be continuous through the adhering material, count as a fiber. Structure must meet AHERA length and aspect ratio criteria. If the structure cannot be observed to be continuous through the adhering material, do not count.

Figure 8   Cluster – Record the length of the longest observable structure. Record width as a best estimate of the average width of the overall structure. The aspect ratio of the overall structure is not a factor. There must be at least three intersections comprised of individual sub-structures that meet AHERA length and aspect ratio criteria to meet cluster definition.

1

2

3

4
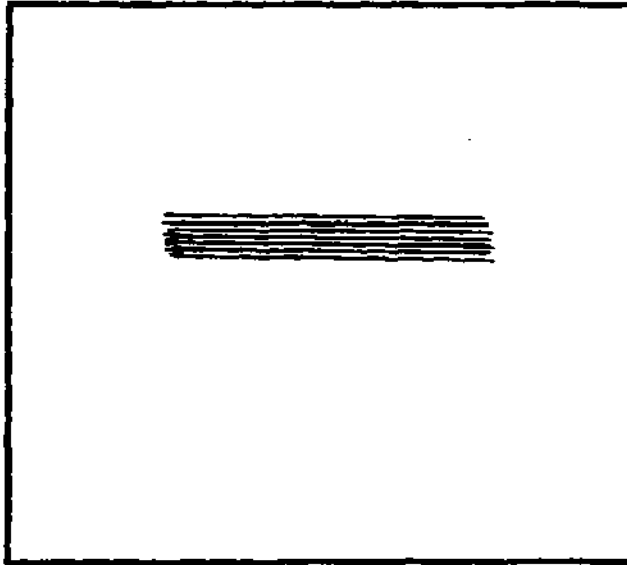
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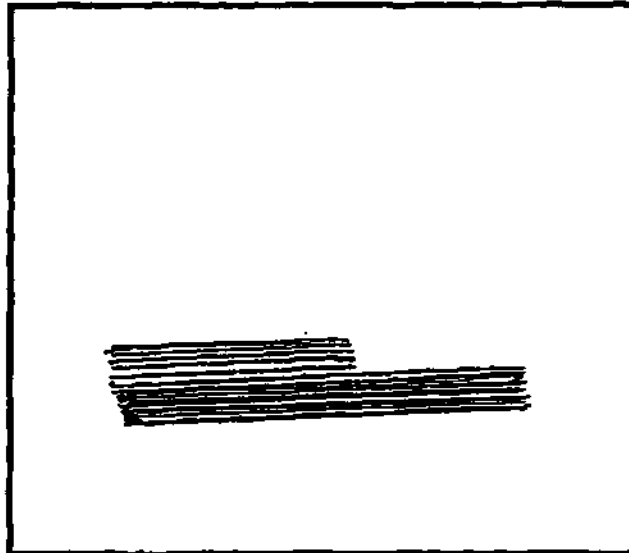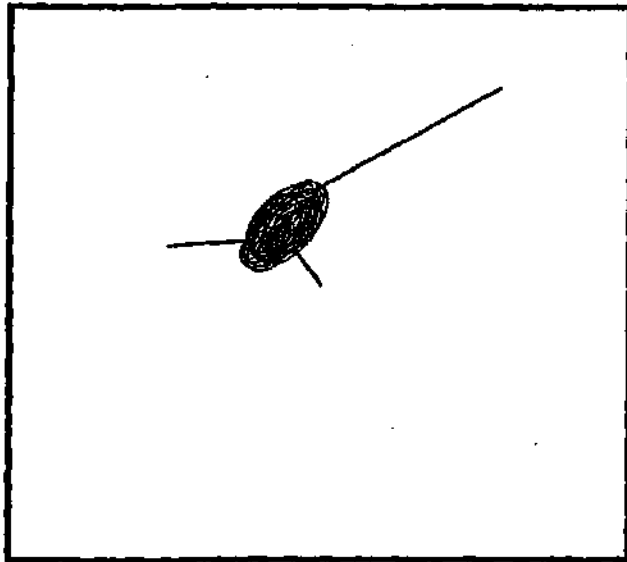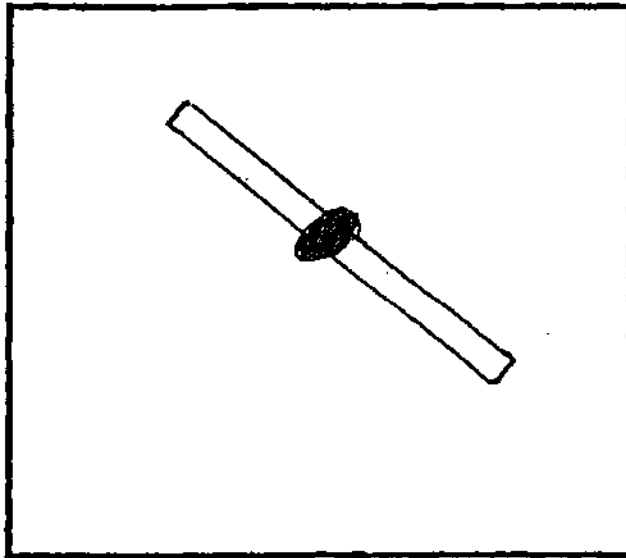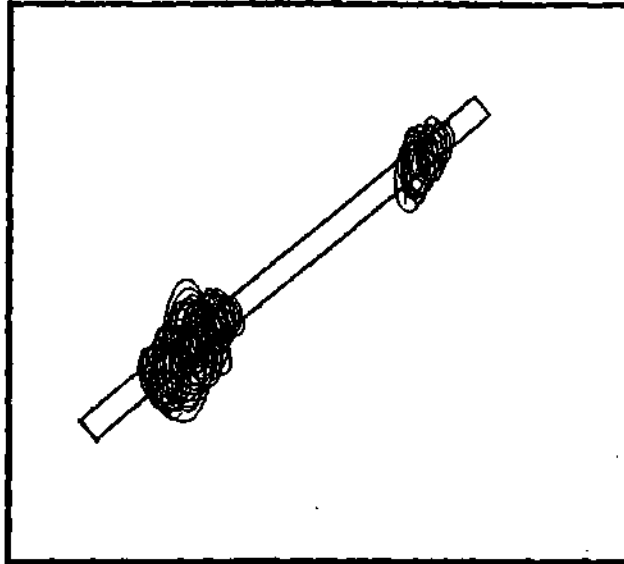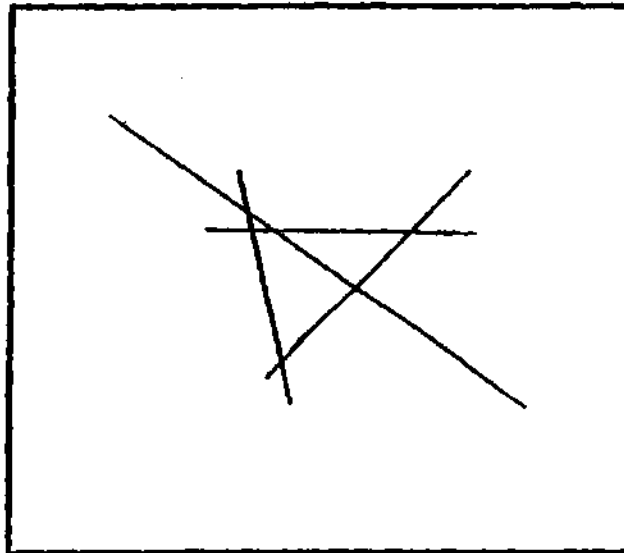

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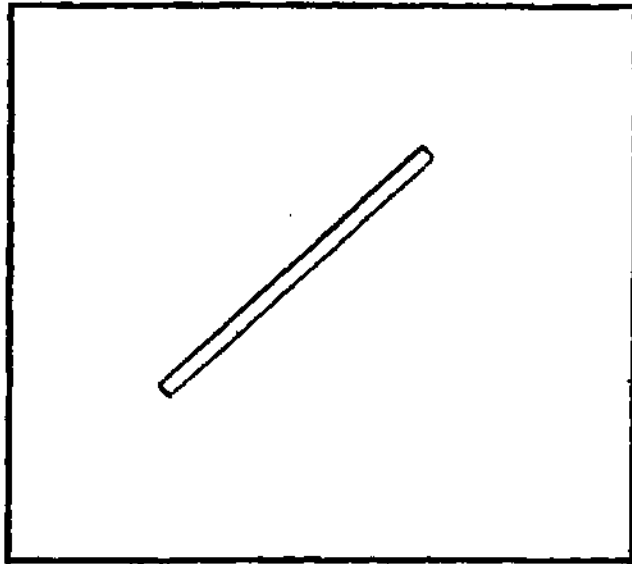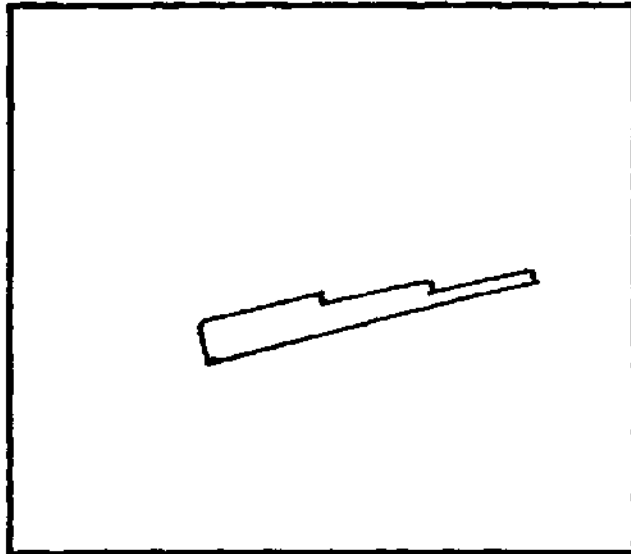
7

8

Request for Modification
To
Laboratory Activities
LB-000030

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin _____ Title: Technical consultant _____

Company: Syracuse Research Corporation _____ Date: 5 August 2003 _____

Description of Modification:

_____ All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance and orientation relative to any nearby landmarks, if present. _____
~morphology,

Reason for Modification:

_____ This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no loner need to be identified before analysis. _____

Potential Implications of this Modification:

_____ There are no potential negative implications resulting from this standardization of QC procedures. _____

Laboratory Applicability (circle one): All   Individual: _____

Duration of Modification (circle one):

    Temporary   Date(s): _____

                  Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

8/14/03

    Permanent   (complete Proposed Modification Section)   Effective Date: (insert based on date of final approval)

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

_____

Technical Review: _W.J. Bratten_____ Date: 8/14/03
            (Laboratory Manager or designate)

Project Review and Approval: _____ Date: 8/14/03
            (Volpe: Project Technical Lead or designate)

Approved By: ___Joey Coddade_____ Date: 8/14/03
        (USEPA: Project Chemist or designate)

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other All other TEM methods, including: SOP EPA-LIBBY-03, SOP EPA-LIBBY-07, and EPA/600/R-94/134 (EPA 100.2).

Requester: W.J. Brattin _____ Title: Technical consultant _____
Company: Syracuse Research Corporation _____ Date: 18 November 2003 _____

Description of Modification:
_____ Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses (all media). _____

Reason for Modification:
_____ This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria. _____

Potential Implications of this Modification:
_____ There are no potential negative implications resulting from this standardization of QC procedures. _____

Laboratory Applicability (circle one): All   Individual: _____

Duration of Modification (circle one):
        Temporary   Date(s): _____
                      Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

        Permanent   (complete Proposed Modification Section)   Effective Date: [insert based on date of final approval] 11/19/03
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

_____

Technical Review: _W.J Brattin_____ Date: 11/19/2003
                  (Laboratory Manager or designate)

Project Review and Approval: _____ Date: 11/20/2003
                  (Volpe: Project Technical Lead or designate)

Approved By: _____ Date: 11/12/2003
                  (USEPA: Project Chemist or designate)

Frequency

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Reprep	1%
Verified analysis	1%
Interlab	0.5%
Total	10%

Each laboratory should prepare and analyze lab blanks, recount (same, different and verified), and reprep samples selected at random in accord with this table. Samples for interlab comparisons will be designated on the COC sheets accompanying the samples.

Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: the procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

Lab Blanks
There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

Re-Analysis.
All re-analysis samples (same, different, interlab, and verified) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 2).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent) For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more errors, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Lab Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

Re-Preparation.
Re-preparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 1. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with ≥1 asbestos structures	0% – 0.1%	0.2% - 0.5%	>0.5%
Recount samples	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrys vs amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Reprep	Concordance on LA count	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 1

STATISTICAL COMPARISON OF TWO POISSON RATES

1.0   INTRODUCTION

An important part of the Quality Control plan for this project is the re-preparation and re-analysis of a number of TEM grids for quantification of asbestos fiber concentrations in environmental media (air, dust, water, soil). Because of random variation, it is not expected that results from re-preparations samples should be identical. This appendix presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0   STATISTICAL METHOD

This method is taken from the textbook entitled "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

$Y1$ = Fiber count in first evaluation
$t1$ = Number of grid openings in first evaluation
$Y2$ = Fiber count in second evaluation
$t2$ = Number of grid openings in second evaluation

The test is performed by following the following steps:

Step 1:

Calculate
$$Y = (Y1+Y2) / 2$$
$$t = (t1 + t2) / 2$$
$$\lambda = Y / t$$

Step 2:

Calculate $Q = (Y1-Y)^2 / (\lambda \cdot t1) + (Y2-Y)^2 / (\lambda \cdot t2)$

Step 3:

Compare Q to the critical value of CHISQ(1-α,1) from the following table:

Alpha	CHISQ(1-α,1)
0.05	3.841
0.10	2.706
0.20	1.642
0.30	1.074

If Q is less than or equal to CHISQ(1-α,1), conclude that the two results are not statistically different at the 100(1-α)% confidence level.

If Q is greater than CHISQ(1-α,1), conclude that the two results are statistically different at the 100(1-α)% confidence level.

ATTACHMENT 2

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos
by Transmission Electron Microscopy-Version 2.0.

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

Shirley Turner
Eric B. Steel

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards
and Technology
Microanalysis Research Group
Surface and Microanalysis Science Division
Chemical Science & Technology Laboratory
Gaithersburg, MD 20899

March 1994

Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysis use the same counting rules.

2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*—transmission electron microscope.

2.1.2 *grid square, grid opening*—an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*—a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information — including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification — is recorded for each observed structure.

2.1.3.1 *Discussion*—Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*—rules used to determine the amount of asbestos present in an asbestos-containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]—procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]—procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*—an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*—a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*—person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*—person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

[1]Code Fed. Reg. 1997, 52 (No. 210), 41826-41905.

[2]ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*—form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*—form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*—the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*—structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*—structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*—To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true positive-unmatched)*—structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*—structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*—the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*—The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*—structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*—false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*—false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*—reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*—reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*—structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*—The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*—a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analyses participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted vs time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS values ≤ 0.05. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

4. Procedure

NOTE 1— This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2— The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3— The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4— The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight forward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5— The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6–4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(Operator\ 1) + TPU(Operator\ 2) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

APPENDIXES

(Nonmandatory Information)

X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____   Date: _____

Grid slot: _____   Verifying Analyst: _____

Grid square: _____

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

*The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1	/	TPU1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1	/	FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	FP1	1	Chr

Analyst 2

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
		P1 / F2			
1	0.1	F1	TPM1	1	Chr
0.8	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr
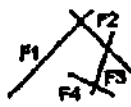
Analyst 2

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

a)

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr

b)

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

c)
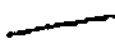
Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
1.3	0.1	/	TPM1	1	Chr
0.6	0.1	‾﹨		1	Chr
1.0	0.1	／	TPM2	1	Chr

Analyst 2

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
1.3	0.1	/	TPM1	1	Chr
1.0	0.1	／	TPM2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to Item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).
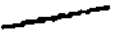
Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNE1 TPM2	1	Chr

b

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr
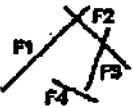
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3			1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr
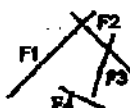
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

Overall Goal: To label all of the reported structures on both count sheets as either TPM, TPU, PP, NL or AMB and to label missed structures as either FNA or FNB.

Compare the two count forms.
Find those structures that match between the two count forms; label matched structures with 'TPM(number)' (4.3)*. An example is given in Fig. X2.1.

Determine if the status of any of the unmatched structures can be unambiguously determined by looking at the count sheets (4.4). Examples are given in Figs. X2.2 – X2.6.

Put the grid in the TEM to resolve the status of any remaining unlabelled structures (4.5). Examples of cases that must be examined by TEM are given in Figs. X2.7 – X2.9. A flowchart for this part of the procedure is given in Fig. X3.2.

Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst.
*Numbers in parentheses in each block refer to the item number in the procedure.

Examine the grid square in the TEM. For each reported structure do the following procedure until all structures are labelled (4.6)*.

Note: If a new structure is identified by the verifying analyst, the structure should be drawn on a new count form and labelled 'TPV(number)'. A label of either 'FNA(number)' or 'FNB(number)' should be put on the two analysts' count forms at the appropriate location (4.6).

Can the reported structure be located (4.5.1)?

Yes →

No → Label the reported structure 'NL' (4.5.1).

Can a judgement be made as to the structure's countability (4.5.2)?

Yes →

No → Label the reported structure 'AMB' (4.5.2).

Is the reported structure countable (4.5.3)?

Yes →

No → Label the reported structure 'FP(number)' (4.5.3).

Optional: If the other analyst sketched the particle and correctly reported it as noncountable, label that analyst's sketch 'TN(number)' (4.5.3).

Is the structure a duplicate of a matched structure on the same count form (4.5.4)?

Yes → Label the sketch 'FP(number)' (4.5.4).

No → Label the reported structure 'TPU(number)' (4.5.5). Switch to the other analyst's count form.

Note: the discrepancy due to counting rule misinterpretation discussed in item 4.4.1 of the procedure is not covered in the flow chart.

Did the other analyst record a sketch of the structure (4.5.6)?

Yes → Label the sketch 'FNA(number)' (4.5.7).

No → Put an 'FNB(number)' on the count sheet at the approximate location it should have been found (4.5.6).

Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other All other TEM methods, including: SOP EPA-LIBBY-03, SOP EPA-LIBBY-07, and EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin _____ Title: Technical consultant _____
Company: Syracuse Research Corporation _____ Date: 25 August 2003 _____

Description of Modification:
_____ Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses (all media). _____

Reason for Modification:
_____ This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria. _____

Potential Implications of this Modification:
_____ There are no potential negative implications resulting from this standardization of QC procedures. _____

Laboratory Applicability (circle one): All   Individual: _____

Duration of Modification (circle one):
        Temporary   Date(s): _____
                        Analytical Batch ID: _____
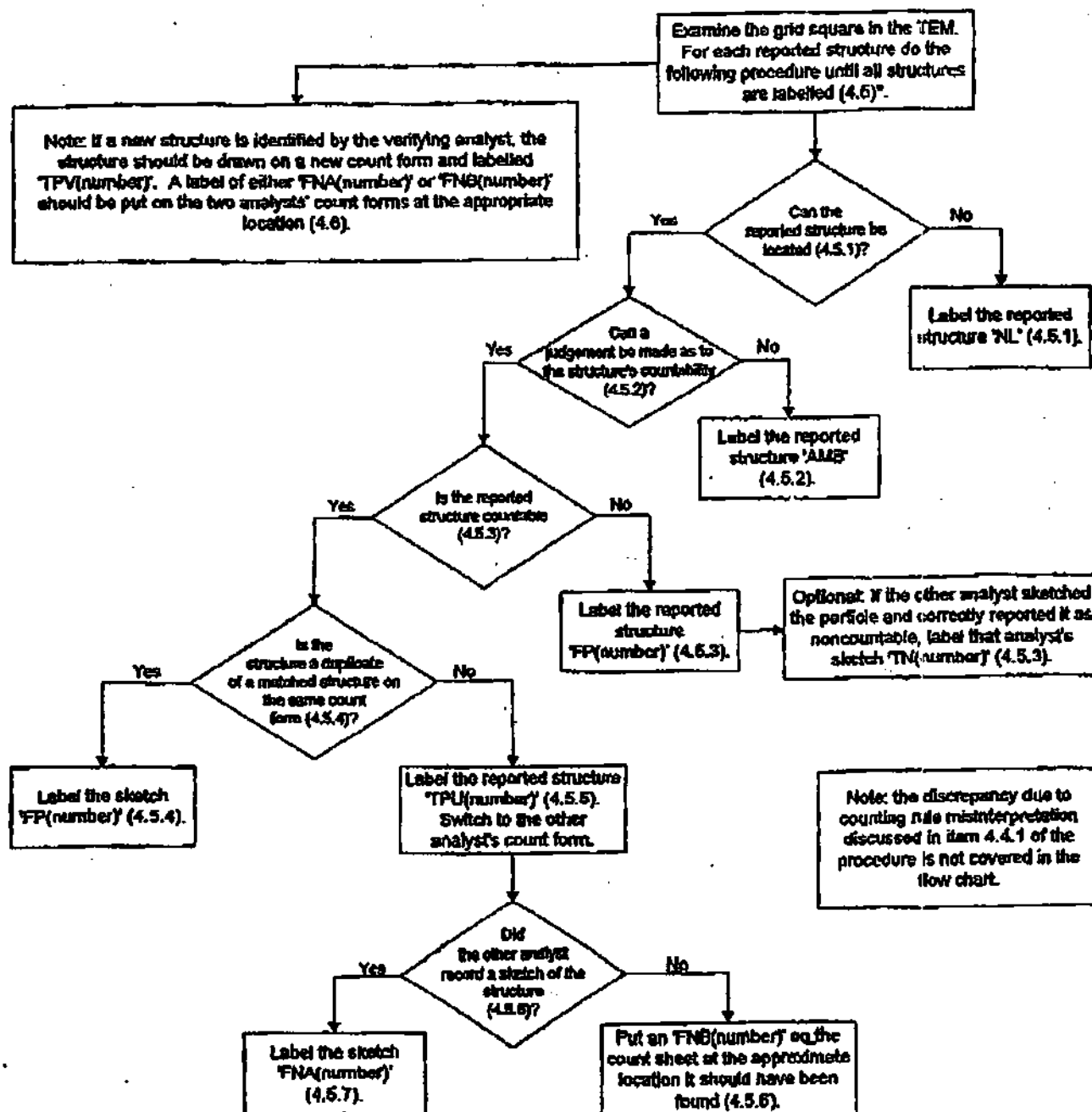Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent   (complete Proposed Modification Section)   Effective Date: (Insert based on date of final approval) 8/26/03
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
_____

Technical Review: _W.J Brattin_____ Date: 8/26/03
            (Laboratory Manager or designate)

Project Review and Approval: _____ Date: 8/27/03
            (Volpe: Project Technical Lead or designate)

Approved By: _____ Date: 8/26/03
            (USEPA: Project Chemist or designate)

Frequency

The frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Reprep	1%
Verified analysis	1%
Interlab	0.5%
Total	10%

Each laboratory should prepare and analyze lab blanks, recount (same, different and verified), and reprep samples selected at random in accord with this table. Samples for interlab comparisons will be designated on the COC sheets accompanying the samples.

Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: the procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

Lab Blanks
There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination.

Re-Analysis.
All re-analysis samples (same, different, interlab, and verified) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. The following criteria will be used to identify cases where results are concordant (in agreement) or discordant (not in agreement). These criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria will be accompanied by scientific justification to support the change.

Measurement parameter	Concordance Rule
Number of asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent) For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more errors, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Lab Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

Re-Preparation.
Re-preparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 1. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

Program-Wide Goals

While each lab shall monitor the results of the QC samples analyzed within their lab and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with ≥1 asbestos structures	0% – 0.1%	0.2% - 0.5%	>0.5%
Recount samples	Concordance on count	>95%	85-95%	<85%
	Concordance on type (Chrys vs amphibole)	>99%	95%-99%	<95%
	Concordance on length	>90%	80%-90%	<80%
	Concordance on width	>90%	80%-90%	<80%
Reprep	Concordance on count	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 1

STATISTICAL COMPARISON OF TWO POISSON RATES

1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the re-preparation and re-analysis of a number of TEM grids for quantification of asbestos fiber concentrations in environmental media (air, dust, water, soil). Because of random variation, it is not expected that results from re-preparations samples should be identical. This appendix presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0 STATISTICAL METHOD

This method is taken from the textbook entitled "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

Y1 = Fiber count in first evaluation
t1 = Number of grid openings in first evaluation
Y2 = Fiber count in second evaluation
t2 = Number of grid openings in second evaluation

The test is performed by following the following steps:

Step 1:

Calculate
$$Y = (Y1+Y2) / 2$$
$$t = (t1 + t2) / 2$$
$$\lambda = Y / t$$

Step 2:

Calculate $Q = (Y1-Y)^2 / (\lambda \cdot t1) + (Y2-Y)^2 / (\lambda \cdot t2)$

Step 3:

Compare Q to the critical value of CHISQ(1-α,1) from the following table:

Alpha	CHISQ(1-α,1)
0.05	3.841
0.10	2.706
0.20	1.642
0.30	1.074

If Q is less than or equal to CHISQ(1-α,1), conclude that the two results are not statistically different at the 100(1-α)% confidence level.

If Q is greater than CHISQ(1-α,1), conclude that the two results are statistically different at the 100(1-α)% confidence level.

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: ___R. K. Mahoney___ Title: _Senior Analyst / Special Projects Coordinator_

Company: ___EMSL Analytical, Inc.___ Date: _17 June 2003_

Description of Modification:

____This is a clarification pertaining to the re-analysis of TEM samples when some of the originally read grid openings in a sample selected for re-analysis have become unreadable. In the event that more than half of the originally read grid openings have become unreadable, select the closest adjacent sample from the same sample delivery group with adequate intact grid openings for re-analysis. If half or less of the original openings on the sample selected are unreadable, make note in the Comments box in Data Entry 1 of the TEM EDD as to which grid openings are unreadable, and proceed with analysis of the original sample.____

Reason for Modification:

____This clarification is intended to provide more complete TEM re-analysis data.____

Potential Implications of this Modification:

____There are no negative implications to this clarification.____

Laboratory Applicability (circle one): [All] Individual(s) _____

Duration of Modification (circle one):

Temporary Date(s): _____

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (Complete Proposed Modification Section) Effective Date: _____17 June 2003_____

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _R. K. Mahoney_ E MSL Date: _18 July 2003_
(Laboratory Manager or designate)

Project Review and Approval: _____ Mark Ravy Date: _7/18/03_
(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: _6/24/03_

Title: _Project Chemist_
(USEPA: Project Chemist or designate)

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other:_____

Requester: R.K. Mahoney _____ Title: Senior Analyst / Special Projects Coordinator
Company: EMSL Analytical, Inc. _____ Date: 25 August 2003 _____

Description of Modification:
This a clarification of laboratory modification LB-000017. If abundant chrysotile is present, the chrysotile count may be terminated at the end of the grid opening in which the 100th chrysotile structure is counted. The analysis will continue recording amphibole structures only until the remaining grid openings to be analyzed are completed. The grid opening designations will be followed by an "*" to indicate the grid openings where only amphibole structures were recorded, i.e. J6*.

Reason for Modification:
This clarification in structure counting and recording is to provide consistency in analytical procedures and data recording in the project laboratories.

Potential Implications of this Modification:
There are no potential negative implications of this clarification.

Laboratory Applicability (circle one): All   Individual(s) _____

Duration of Modification (circle one):
    Temporary   Date(s): _____
                  Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

    Permanent   (Complete Proposed Modification Section) Effective Date: _____ 23 January 2002 _____
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: _R.K. Mahoney EMSL_ _____ Date: 3 September 2003
                  (Laboratory Manager or designate)
Project Review and Approval: _____ Volpe _____ Date: 9/8/2003
                  (Volpe: Project Technical Lead or designate)
Approved By: _____ Jay Goddard _____ _____ Date: 8/25/03
    Title: _Project Chemist_ _____
                  (USEPA: Project Chemist or designate)

Lab Modification Form Revision 5

Appendix 3

- EMSL Analytical, Inc. - Standard Operating Procedures, Asbestos Analysis, PLM Analysis of Bulk Samples, 8/1/00

- EMSL Analytical, Inc. - Standard Operating Procedures, Quality Control Program, Asbestos Laboratory, 2/01

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STANDARD OPERATING PROCEEDURES FOR

EMSL ANALYTICAL INC.

POLARIZED LIGHT MICROSCOPY

Standard Operating Procedures
Asbestos Analysis
PLM Analysis of Bulk Samples

Revision Date:	**July, 2000**
Original Date:	**June, 1995**

Issue Date: August 1, 2000

EMSL Analytical Inc., Quality Assurance Dept.

EMSL ANALYTICAL

**Standard Operating Procedures for
the Analysis of Asbestos by
Polarized Light Microscopy**

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I

LOG-IN, REPORTING AND SAMPLE HANDLING

1.0 INTRODUCTION

Standard Operating Procedures (SOPs) documented in this manual are intended for information and use of all laboratory personnel employed by EMSL, Inc. at all laboratories that perform asbestos analysis of bulk material by Polarized Light Microscopy. Currently, PLM analysis is performed at all EMSL laboratories. The Corporate Quality Assurance Department is responsible for the control and oversight of any revision in these Standard Operating Procedures. All laboratory personnel must adhere to procedures documented in this manual. Any major deviations must be approved by the Quality Assurance Department. Standard Operating Procedures for all other analysis are presented in other SOP's.

2.0   SAMPLE RECEIPT/LOG IN PROCEDURES

Care should be used to avoid cross contamination of bulk samples and air samples when handling incoming samples.

Acceptance of Bulk Samples

Incoming samples are inspected by the Sample Receiving Coordinator to determine if they conform to laboratory acceptance criteria.   Samples are judged unacceptable under the following circumstances:

- Bulk samples must not be packaged with air samples
- Samples must not be submitted in obviously damaged or compromised packaging
- Analysis requested must not be outside laboratories capability or accreditation
- Sufficient amount of sample must be provided.
- The sample identification must correspond to those listed on the client's Chain of Custody form
- Requested turnaround must be achievable
- Proper sampling technique must have been performed
- Appropriate analytical methodology must be cited

Samples, which do not meet the above requirements, are rejected. Deficiencies are reported to the client and noted on the Chain of Custody in the comments section of the form. A deficiency/corrective action report is completed and filed.

Samples are accepted upon signing the chain of custody and logged into the Laboratory Information Management System (LIMS), as described in the following section.

2.1   Verification of Chain of Custody for Bulk Samples

All samples received must be submitted with a Chain of Custody Form. An EMSL Chain of Custody form is available for clients who do not have one. The Chain of Custody form contains information regarding samples, which is essential for proper analysis, sample tracking, and record keeping. All facilities must verify the information contained on this form prior to the log-in procedure, beginning with sample sets with the quickest turnaround time.

The Chain-of-Custody Form must be checked for the following:

- The sample numbers on the chain of custody form must match the numbers on each sample exactly.
- Each sample received must be listed on the Chain-of-Custody form.
- The turnaround time should be clearly stated. In the case of a regular client, these may be known in advance. If the turnaround time is not known or is incorrect, the client must be notified immediately.
- Contact information for reporting of results or for addressing any questions or problems, is included on the Chain-of-Custody form.
- The person relinquishing the samples must sign where appropriate.
- If the client wishes to make changes i.e., turnaround time or type of analysis, the client must provide such information in writing. If possible, the client will send the written statement by facsimile.

2.2   Assigning Laboratory Numbers and Sample Log-In

After this information has been verified, each sample to be analyzed may now be entered into the computerized log in system and assigned a unique laboratory number. Instructions on Computer Data Entry can be found in the LIMS Computer Manual.

2.3   The Following Information Is Recorded When Samples Are Logged In:

- Client Name and Address
- Job Number or Name of Project (or both if available)
- Quantity of Samples Ordered
- Type of Analysis
- Turnaround Time
- Sample ID
- Phone Number
- Results to:
- Fax #
- Purchase Order # (if available)
- Name of individual logging samples

The computer then assigns a laboratory number to each sample.

Voided samples

If at any time during acceptance, log in, or analysis procedures a sample or sample set is voided for any reason, the laboratory must document such an event. This includes a notation on the Chain of Custody and record on the Deficiencies and Corrective Action Report (EMSLdfcorr1.98).

Samples may be voided by the client or the Laboratory Manager. Examples of causes may be:

- Work order cancelled by the client
- Analysis requested outside laboratory capability
- Obviously damaged or compromised samples, i.e. opened air cassettes, cassettes with torn or ripped filters, water samples in leaking or faulty containers.
- Obvious faulty sampling technique
- Improper sample media
- Incompatible samples packaged together (i.e.- air samples with bulk samples)
- Inappropriate analytical methodology requested

The client must be immediately notified by the laboratory manager or designee when the sample is voided.

2.4 Required Documents

After samples are assigned a unique laboratory number and have been logged in, additional data and special forms are generated for each sample set.

Forms are generated either by entering data into the computerized log in data system (see the LIMS Computer Manual) or manually, depending on the specific form.

Billing Worksheets, PLM Analysis Worksheets, and Tracking Labels are generated for each set of samples.

2.5 Billing Worksheet

The Billing Worksheet is produced by computer after sample information has been entered and samples have been assigned a unique laboratory number. Instructions for producing a computerized Billing Worksheet are in the Computer Manual.

The Billing Worksheet contains the following information:

- Computer Billing #
- Client Name and address
- Project ID
- Turnaround Time
- Type of Analysis
- Quantity
- Date
- Laboratory #'s
- Sample ID and location
- Purchase Order #

- Accounting code
- Phone #, Fax #
- Logged in by and time
- Prepared by and Date
- Analyzed by and Date
- Data entered in by and Date
- Screened by and Date
- Mailed out and Date
- Comments
- Laboratory location

2.6 PLM Analysis Worksheets

Analysis Worksheets are generated by the computer at the time of log in. Specific instructions for preparing a computerized PLM Analysis worksheets are found in the LIMS manual.

The analysis worksheet contains the following information:

- Client information (address, phone, etc.
- Time logged in
- Date/time Due
- Project Identification (if provided)
- Turnaround time
- Billing number
- Client sample identification number
- Location from which the sample was collected
- Appearance of sample
- Treatment
- % and type of asbestos
- % type of non-asbestos
- optical properties of non-asbestos
- Optical properties of asbestos
- Analysts signature, date

3.0   BULK SAMPLE PREPARATION – OVERVIEW

Bulk samples are prepared under a HEPA filtered hood in an area separate from air samples.   Technicians are instructed in basic sample preparation techniques, instrument calibration and safety procedures.   After receipt and verification of all paperwork as specified in Sample Receipt, Section 2.0, the following steps are taken:

Open the sample container under the HEPA filtered hood and place entire contents on a clean sheet of paper.   In the event that the sample size is too large to fit onto the paper, make a composite sample with several representative portions of the sample and place them on the sheet.   Observe the sample under the stereomicroscope; examining for homogeneity, color, and obvious fibers.   Determine the appropriate category for sample preparation technique, which should be employed, i.e. crushing, teasing, dissolving or other mechanism and estimate as much as possible the percentage of all material present (see Section II).   Note on the PLM Analysis Worksheet the sample color, method of treatment used, and degree of homogeneity.

Preparation Techniques

Teasing

Take two pairs of cleaned forceps and hold the sample down with one of the forceps. Use the other pair of forceps to pull and tease the sample material apart. If curly fibers are present, place a drop of 1.55 RI liquid on a pre-cleaned glass slide.   Grasp the sample material with the forceps and place it into the liquid on the slide and cover with a coverslip.   Flatten material between the coverslip and slide by gently rubbing a pencil eraser over the slipcover.   If straight fibers are observed, indicating the potential presence of amphiboles, mount the sample in 1.68 RI liquid and follow the same procedure as above.   If both curly and straight fibers are observed, prepare multiple sample preparations as discussed above.   If no fibers are observed, mount in 1.55 RI liquid.

Crushing

Use forceps or other appropriate instrument that has been cleaned to isolate a small, representative piece and scrape or crush the material.   If curly fibers are present take a drop of 1.55 RI liquid and place on a pre-cleaned glass slide. Grasp the sample material with the forceps and place into the liquid on the slide and cover it with a coverslip.   Flatten the material between coverslip and slide by rubbing a clean pencil eraser over the coverslip.   If straight fibers are observed, mount the sample in 1.68 RI liquid and follow the same procedures as above.
If both curly and straight fibers are observed, prepare multiple sample preparations as discussed above.   If no fibers are observed mount in 1.55 RI liquid.

Dissolving

Isolate a small representative sample using cleaned scalpel and forceps. Place the isolated sample on a pre-cleaned slide and dissolve with the appropriate solvent (i.e., acetone, chloroform, tetahydrofuran, toluene, and amyl acetate). Allow the solvent to evaporate before addition of RI liquid (air evaporation, or hot plate evaporation).

Note: Perform this preparation technique under a fume hood suitable for organic compounds. See EMSLCHP100.0

If curly fibers are present mount the sample in 1.55 RI liquid and cover with a coverslip. Flatten the material between coverslip and slide by rubbing a clean pencil eraser over the coverslip. If straight fibers are observed, mount the sample in 1.68 RI liquid and follow the same procedure as above. If both curly and straight fibers are observed prepare multiple sample preparations as discussed above.

Carefully fold the sheet of paper used during stereoscopic review and dispose of in designated as asbestos waste receptacle.

4.0 ANALYSIS OF BULK SAMPLES – OVERVIEW

PLM Analysis Procedure

After a sample has been prepared, observe under the polarized light microscope. All fibrous materials are identified. If amphiboles are suspected, preparations are made consecutively in 1.68 and 1.605 liquids to determine the refractive index of the fiber. Observations of optical properties of suspect asbestos fibers are made and recorded to include:

- morphology
- pleochroism (if any)
- retardation
- thickness (as applicable to determine birefringence)
- refractive index parallel and perpendicular to polarizer
- sign of elongation
- angle of extinction
- color of fiber

Optical characteristics of suspect non-asbestos fibers are also recorded. These may include:

- Isotropic
- Undulating
- High Birefringe
- Scaly surface
- Double sign of elongation

An area percent of asbestos is determined by calibrated visual estimation or by point count criteria. Record all information obtained during analysis the PLM Analysis Worksheet.

5.0 DATA REPORTING

Procedures for Reporting Data

Following sample analysis, the analyst initials the billing worksheet and signs the analyst worksheet containing the sample results. The analyst then submits the results for data processing. Following data entry and report generation, the report is submitted for review and signature by the laboratory manager or designee. Procedures for report clearance includes:

- Overall compliance with the Quality Assurance Program
- Check of daily calibrations including standard analysis
- Review of intra analyst QC data associated with sample set
- Comparison of original data with typed report
- Check of completeness of bench forms

Approved signatories are assigned by the Regional Laboratory Managers or the Quality Assurance Manager.

- The billing worksheet is signed and dated by appropriate personnel.

Report filing

Files are maintained of the original bench worksheets, billing worksheet, copy of the chain of custody and any other applicable associated paperwork. All documents are filed by billing number.

6.0 DATA ENTRY AND THE FINAL REPORT

Data produced during analysis is entered into the computer. Specialized computer programs corresponding to different types of analyses have been written. Instructions for entering data are kept in the LIMS Computer Manual.

All reports must be signed by an approved signatory and will contain the following disclaimers:

> "The above test report relates only to the items tested. This report may not be reproduced, except in full, without written approval by EMSL. The above test must not be used by the client to claim product endorsement by NVLAP nor any agency of the United States Government".

> "Laboratory is not responsible for the accuracy of results when requested to physically separate and analyze layered samples"

Verification of Data

After the report is printed, the data entry operator reviews all reports as follows:
- Correct client information appears on the report.
- Sample numbers are correct
- Checks for any obvious errors
- Sample components sum to 100%

Assembling Reports

Place the pages of the report, the original bench worksheets and all other project related documents together for final review by the laboratory Approved Signatory.

Signing the Final Report

Prior to signing, the printed final report with all its components is reviewed in detail by an approved signatory. The report is reviewed for:

- Overall compliance with the Quality Assurance Program
- Typographical and transposition errors, which may occur between the client chain-of-custody form, analysis worksheets and final report
- All required signatures or initials are present in the Analysis Worksheets.
- Review of microscope calibration records and intra analyst QC data
- Review of original data for technical accuracy and completeness
- Check of daily calibrations including standard analysis

When the report review is complete, the approved signatory signs the report in the space required and places the documents in the 'to be mailed' bin. Reports containing errors or lacking information are held back for corrections by the data entry staff.

Final reports are mailed by the clerical staff under the direction of the Laboratory Manager.

7.0   FILING SYSTEM

After final report is sent to the client, the billing worksheet, original bench worksheet and a copy of the chain of custody is filed by billing number.   Final reports are maintained electronically by the LIM's system. Copies of chains of custody are stored at the corporate office in computer files.
All records are stored for 7yrs.

8.0   SAMPLE STORAGE AND DISPOSAL

All samples are placed in ziplock bags and kept for one month (unless otherwise requested by the client). Samples containing <1% asbestos are discarded into the trash while those containing $\geq$1% asbestos are discarded through a licensed hazardous waste removal company. A copy of the waste manifest is stored in the laboratory files.

9.0 PROCEDURE FOR DEALING WITH CLIENT COMPLAINTS OR PERFORMANCE DEFICIENCIES

If a client calls with a complaint, the call is referred to the Laboratory Manager.   An investigation into the cause of the problem is initiated. The Laboratory Manager will review all applicable documentation related to the sample including bench notes, chain of custody and Quality Control data. The Laboratory Manager may need to discuss the analysis with the responsible analyst. When the discrepancy is resolved, the client is notified and a corrective action report is completed and placed in the file. In the event a resolution is not accomplished, the Corporate QA Manager is called upon to aid in satisfying the issue.

A client complaint or performance deficiency may be defined as:

- Reporting errors such as typographical errors, transcription errors
- Missed turnaround time requirements
- Analytical or technical errors
- Sample numbering
- Results reported to wrong client contact
- Miscalculated results

Steps for resolution may include:

- Additional training-This may include one-on-one training, refresher workshops, etc.
- Revisions to report review procedures
- Adding to laboratory resources (staff, equipment, etc.)

If the problem has not been resolved, and involves a result discrepancy, it is EMSL's policy to suggest the sample(s) be analyzed by another laboratory. If a discrepancy is still found, the samples will be sent to a third, referee laboratory. The laboratory should be selected and agreed upon by EMSL and the client.

For each event, a deficiency corrective action form (EMSLdfcorr1.98) must be completed and maintained in the laboratory files.

II

DETERMINATION OF ASBESTOS IN BULK SAMPLES BY POLARIZED LIGHT MICROSCOPY (PLM) WITH DISPERSION STAINING

6.0   QUALITY CONTROL

6.1   Instrument Calibration/Maintenance

Follow the manufacturer's instructions for illumination and condenser alignment and other microscope adjustments. Specific guidelines for calibration, alignment, and maintenance can be found as Appendices in this manual.

Maintenance is a daily activity and is the responsibility of each analyst. Preventive maintenance is emphasized. Repairs beyond the capabilities of the analyst must be referred to an outside vendor.

Alignment/calibration must be checked weekly or more frequently, especially if the microscope is transported. See Calibration Frequency Reference Guide in Appendix section of this manual for a detailed schedule of calibration frequencies.

6.2   Personnel Training and Qualifications

Technicians hired for PLM analysis, which have not had previous experience or formal training, require the greatest amount of training and attention. They are introduced to basic information prepared by our more experienced analyst and are simultaneously led through the basics in the prep room and at the microscope. The trainee will work very closely with the mentor, helping and learning but not analyzing alone. Following this initial training period, the trainee analyzes samples, which are also run by the mentor. Through this activity, the progress of the trainee can be gauged without endangering the quality of data being reported by the lab. If the progress is good and the mentor feels confident about his/her student, an attempt is made to allow the candidate to run the QC samples, both the re-analysis and re-preps re-analysis. The trainee will be deemed proficient when quantitation and qualification within laboratory norms as established by our QC program is demonstrated on 100 consecutive samples. Additionally, the trainee must perform analysis on five rounds of past proficiency samples and succeed in generating data with the acceptable range as established by the agency(ies) statistical analysis.

Using NIST Standard Materials, the analysts must also be able to demonstrate ability to measure optical properties of asbestos fibers. The analyst must be able to determine the properties of all six asbestos types.

During this stage, the training analyst is trained and tested in textbook theory on microscopy and optical mineralogy. The analyst continues to receive extra attention and coaching as he/she analyzes on a microscope physically situated near that one operated by the mentor. Furthermore, whenever possible, the novice analyst is encouraged to participate in continuing education workshops and in-house programs related to PLM analysis. An analyst who has received the above training and attention and who consistently performs well will be considered qualified. Qualification status is determined by the Laboratory Manager or Quality Assurance Manager.

6.3   Intra-Analyst Reanalysis/Quality Assurance

Intra-analysts analysis is performed on 2%, or 1/50 of the samples analyzed daily (at a minimum). These samples are randomly chosen by the laboratory analyst. The same sample is prepared and analyzed a second time by the same individual. This data is then entered into the monthly Quality Control program.

6.4   Inter-Analyst Quality Assurance

Inter-analysts analysis (by at least one other analyst) must be performed on 7%, 1/15 of all samples analyzed. Samples are chosen randomly. Each analyst will have no knowledge of the other analyst's (original) results prior to his/her evaluation. This data is entered into the monthly Quality Control program.

Non friable organically bound (NOB) samples-reprep
Complete repreparations of NOB samples shall be performed on 5% or 1 in 20 for interanalyst QA. This includes all steps for gravimetric reduction .

6.5   Inter-Laboratory Quality Assurance

Inter-Laboratory analyses are samples that are reanalyzed by another laboratory. This process evaluates the precision of the participating laboratories preparation and analysis procedure.

Each EMSL laboratory will exchange samples with another laboratory on 1 in 500 samples and at a frequency of 4 times a year. The Regional Manager is responsible for maintaining and managing the program. The data is submitted to the Quality Assurance Department in the Monthly Quality Control Report.

1.0 OVERVIEW

1. This method describes the procedures for the determination of the presence or absence of asbestos in bulk samples of building material. Samples are initially examined under low magnification using a stereo microscope, contained in a hood equipped with a HEPA filter. Initial observations should note gross material appearance (homogeneity, fibrous/non-fibrous) and physical characteristics (color, texture, friable/non-friable).

2. Analysis by polarized light microscopy (PLM) is used for the positive identification of suspect fibers. Positive identification of asbestos requires the determination of several optical properties peculiar to the six types of asbestos: chrysotile asbestos, grunerite asbestos (amosite), riebeckite asbestos (crocidolite), anthophyllite asbestos, tremolite asbestos and actinolite asbestos.

3. Quantitative estimates of the asbestos content, and other major constituents, of the sample are made based on a combination of the estimates from both the gross and the PLM examinations.

4. Interference's from other inorganic and organic fibrous constituents, cleavage fragments of natural minerals, binders, coatings, and man-made fibers may be encountered. Moisture may interfere with the determination of some optical properties. Therefore, wet samples should be dried prior to analysis.

5. The sample matrix may cause a variety of interference's under PLM observation. Special matrix reduction techniques may be necessary to reduce these interference's.

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2.0   EQUIPMENT

1.   A low power binocular microscope (preferable stereomicroscope), with a magnification range of approximately 10-45X, and an auxiliary light source.

2.   A compound microscope set-up for polarized light microscopy, to include a polarizer, analyzer, port for a wave retardation plate, a 360° graduated rotating stage, substage condenser, lamp and lamp iris.

- Objective Lenses: 10X, 20-25X, 40-45X, and dispersion staining objective.
- Ocular Lens: 10X minimum
- Eyepiece reticule: Cross hair
- Compensator plate: 550 millimicron retardation (first-order red or gypsum)

3.   The type of material being examined will dictate the various apparatus needed for sample preparation. At a minimum, the following will be required:

- Negative pressure hood equipped with a HEPA filter at the exhaust
- Microscope slides: ~75 mm x 25 mm, 1 mm thickness
- Coverslip: No. 1, 22 mm^2
- Tweezers, tungsten probes, dissecting needles, scalpels, glazing pliers, forceps.
- Glass plates, petri dishes or disposable containers(e.g. weighing boats 5^{-2})
- Mortar and pestle (agate or porcelain)

4.   Auxiliary equipment may include a Wylie mill, centrifuge, filtration apparatus, and low temperature ashers, assorted beakers, and miscellaneous glassware, a vacuum cleaner equipped with a HEPA filter.

3.0 REAGENTS

1. Refractive index liquids

    - N_D = 1.550, 1.605, 1.630, 1.680, 1.700

2.

    - Dilute acetic acid (CH_3COOH): ACS reagent grade
    - Dilute hydrochloric acid (HCl): ACS reagent grade
    - Acetone (CH_3COOH_3): ACS Reagent grade
    - Chloroform ($CHCl_3$): ACS Reagent grade

3. Asbestos reference standards, and standards for various minerals and man-made materials typically encountered in bulk materials containing asbestos. Use NIST Certified SRM 1866a/Common Commercial Asbestos, SR1867/Uncommon Commercial Asbestos.

4.0   BACKGROUND AND DEFINITIONS

The name asbestos, a Greek word mistakenly thought to mean incombustible, was given to fibrous minerals hundreds of years before the science of mineralogy evolved. The Greek word actually means unquenchable, inextinguishable (not incombustible) according to the etymology of the Oxford English Dictionary.

The definition of asbestiform minerals includes three aspects: morphology, structure, and chemistry. Morphologically, asbestiform mineral varieties separate into flexible fibers or flexible bundles of fibers. Flexible fibers bend readily and only break across the fibers into distinct pieces with some difficulty. Structurally, the asbestiform minerals are limited to the serpentine and amphibole mineral groups. Chemically, these minerals are all hydroxylated silicates. The term "hydroxylated" is preferred over "hydrated" because these minerals contain OH ions rather than water or crystallization. The serpentines contain approximately 13-weight percent water; and the amphiboles, approximately 2.5 weight percent water.

There is no "group" of asbestos minerals. "Asbestos" is a general term applied to certain minerals (which are themselves classified under crystal-structure-based groups) when these minerals crystallize as the asbestiform variety. Table 2 lists some common silicate minerals and their asbestiform varieties, together with their relationships and formulas in Tables 3 & 4.

Only very small quantities of the amphibole and serpentine minerals under particular geological circumstances occur as an asbestiform variety of the mineral. The asbestiform varieties occur in veins or small veinlets within rock containing or composed of the common (nonasbestiform) variety of the same mineral.

In some rare instances, the mineralogical occurrences contain sufficient quantities of usable asbestiform minerals to be economically mined for commercial asbestos. The soft, silky fibers of asbestos (sometimes called mineral silk) are so flexible that they can be spun into threads from which cloth can be woven. The resulting material is fireproof, is a good thermal and electrical insulator, and has moderate to good resistance to acids. It has been used from Roman times, and is most familiar in daily use in brake lining for automobiles and as the "asbestos" siding used in residential construction.

The six asbestos minerals are defined under two mineral groups:
1. The serpentine group and
2. The amphibole group.

Serpentine Asbestos

Chrysotile is the only commercial asbestos mineral belonging to the serpentine group. Moderate amounts of aluminum may substitute for silicon and moderate amounts of iron may substitute for magnesium. Small amounts of manganous oxide (Mn)), calcium oxide (CaO), potassium monoxide (K_2O) and sodium monoxide (Na_2O) are also reported in the chemical analyses.

The crystal structure of chrysotile asbestos consists of double layers. Each layer consists of a linked SiO_4 tetrahedral coordinated to a second layer of linked MgO_2 $(OH)_4$ octahedral through a sharing of oxygen atoms; the composite double layer rolls up (like a window shade) to form long hollow tubes. The diameters of the individual tubes are on the order of 35 mm, and the length-to-diameter ratio can vary from 10:1 to well over 10,000:1.

Chrysotile is characterized by a combination of (1) a distinctive shape, (2) a chemical composition close to $Mg_3Si_2O_5$ $(OH)_4$, and (3) characteristic X-ray and electron diffraction pattern.

Amphibole Asbestos

Five of the six commercial asbestos minerals belong to the amphibole mineral group. These are grunerite asbestos (usually but improperly referred to by the acronym amosite); riebeckite asbestos (usually referred to by the variety name crocidolite); anthophyllite asbestos, tremolite asbestos; and actinolite asbestos. A considerable amount of substitution of other elements for Fe^{2+}, Fe^{3+}, silicon, sodium, calcium, and magnesium can take place in these minerals.

The Crystal structures of the amphibole minerals, including the asbestiform varieties, are composed of strips or ribbons of linked polyhedra, which join to form the three-dimensional crystal. The individual stripes are composed of three elements: These are two double chains of linked (Si, Al)O_4 tetrahedral and a strip of linked MgO_6, FeO_6 or AlO_6 octahedral.

4.1 Properties of Asbestos

Asbestos is a fibrous mineral of unique properties. It is used in a multitude of different applications because it can confer superior properties on products, including the following:

- stability in resistance to heat, moisture and microorganisms;
- insulation against noise, heat and electricity
- resistance to wear and to deformation under load or impact
- improved smoothness, hardness and opacity
- resistance to chemical attack, leaching and decay.

4.2 Asbestos Related Terms

In the following discussion, asbestiform refers only to asbestos. The other term, "fibrous", "mineral fiber", "fibril" and "fibril structure" applies to both asbestiform and non-asbestiform varieties.

Asbestos: A collective mineralogical term encompassing the asbestiform varieties of various minerals; an industrial product obtained by mining and processing primarily asbestiform minerals.

The quality of asbestos depends on the mineralogy of the asbestiform variety, the degree of asbestiform development of the fibers, the ratio of asbestiform fibers to acicular crystals of other impurities, and the length and flexibility of the fibers. The major asbestiform varieties of minerals used for asbestos are chrysotile, tremolite-actinolite asbestos, cummingtonite-grunerite asbestos, anthophyllite asbestos, and crocidolite. Asbestos may be marketed by its mineral name such as Amosite or Montasite. Some asbestos products contain non-asbestiform minerals (for example, asbestos-cement and asbestos-magnesia); consequently, the mineralogical and the industrial definitions of asbestos do not always coincide.

Fibrous: The occurrence of a mineral in bundles of fibers, resembling organic fibers in texture, from which the fibers can usually be separated (for example, satin-spar, and chrysotile).

The term "fibrous" has been used during the last 200 years to describe all kinds of minerals that crystallized in habits resembling organic fibers, including asbestos minerals. However, the related term "asbestiform" was never used for fibrous mineral habits other than asbestos. Accordingly, "fibrous" is the more general term, and asbestiform is a specific type of fiber.

<u>Mineral Fiber</u>: The smallest elongated crystalline unit which can be separated from a bundle or appears to have grown individually in that shape, and which exhibits a resemblance to organic fibers. (Examples: fiber bundles, chrysotile and crocodile, individual fibers, epsomite and Millerite).

The term "fiber is not limited to asbestos. However, it is distinct from "acicular" because it requires the resemblance to organic fibers.

<u>Fibril</u>: A single fiber, which cannot be separated into smaller components without losing its fibrous properties or appearances.

Most fibers are single structural entities, such as Millerite and nickel sulfide, and some may be called fibrils. However, some fibers are composed of two or more fibrils that are less readily separable from each other than fibers are from bundles (for example, chrysotile and crocidolite).

<u>Fibril Structure</u>: A systematically deformed and/or defective crystal structure of a fibril. A defect structure would involve various type of dislocation. The fibril structure may be exhibited by a single crystal, a group of single crystals, or at twinned single crystal.

The scroll-like fibril structure of chrysotile, the twinned single crystal fibrils of chrysotile, and the incompletely resolved fibril structure of an amphibole are all examples illustrated in the literature.

Some acicular single crystals may have the appearance of fibers and fibrils, yet there is nothing unusual about their crystal structures. Other acicular single crystals may have significant structural deviation sin addition to appearance which result in the display of certain properties usually found in fibers such as high tensile strength along the fiber axis. Thus, fibril structure is not limited to asbestiform structures, but may occur in a minor form in non-asbestiform structures.

<u>Asbestiform</u>: A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

"Asbestiform" and "asbestos" are essentially synonymous in current usage. Some special properties of asbestiform varieties, including optical extinction and surface charge, are either not fully understood or are not uniformly applicable to all asbestiform fibers; consequently, they cannot be considered fundamental characteristics at this time.

4.3 Commercial Asbestos Minerals

Chrysotile: found in white, wavy, silky, lustrous fiber bundles. The fibers are usually much longer than they are wide. Chrysotile is often found in woven materials because of its flexibility.

Amosite: found in tan-brown, straight, brittle, rigid, inflexible fiber bundles.

Crocidolite: found in blue-blue-gray, straight, rigid fiber bundles. It is probably the most toxic form of asbestos we know.

Anthophyllite: usually colorless to pale brown. It may be found as singly crystals or fiber bundles. Fibrous anthophyllite is generally long and thin.

Actinolite and tremolite: difficult to identify, appear as acicular (bladed) and prismatic (more massive) cleavage fragments.

4.4 Polarized Light Microscopy Terminology

Crossed polarized (polarizer and Analyzer crossed): A fiber is isotropic (has only one refractive index) if it appears black (dark on a dark background) as the stage is rotated. It is extinct at all angles. Such a fiber cannot be an asbestos fiber.

A fiber is anisotropic (has more than one refractive index) if it shows up, as the stage is rotated, alternately light on a dark background

Sign of Elongation: A first order red plate is a section of quartz. It produces a 530nm retardation between the fast ray (X' along the long edge of the plate) and the slow ray (z' along the short edge of the plate). At crossed polars, if the fiber turns yellow in a NW-SE direction (parallel to the red plate port), it displays a positive sign of elongation. If the fiber turns blue when oriented in a NW-SE direction, it displays a negative sign of elongation. Crocidolite is the only asbestos mineral with a negative sign of elongation.

Dispersion Staining: λo is the wavelength at which solid and liquid match in refractive index. Dispersion staining requires "stops" in a special objective. The annular stop allows colors through. The central stop allows complementary (white light -λo) colors to pass through. Reference tables exist which show the complementary annular and central stop colors for different asbestos minerals in different immersion liquids. If fiber and liquid RI's are too far apart, then no dispersion staining colors will result.

Pleochroism: Pleochroism is one of the least reliable asbestos identification characteristics. Pleochroism refers to the tendency of a fiber to change color tint when rotated on the stage in plane polarized light. Most asbestos minerals are nonpleochroic. That is, they do not appear to change color tint as the stage is rotated in plane polarized light. Filler-binder materials contained in the insulation sample, however, may coat the asbestos fiber bundles and create a false pleochroic response. The most strongly pleochroic asbestos mineral is crocidolite, which usual appears to change from a blue to a blue-gray as the stage is rotated.

5.0   ANALYTICAL METHODOLOGY

Note : Exposure to airborne asbestos fibers is a health hazard.   Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuos airflow (negative pressure) and a HEPA filtered exhaust. Handling of samples without these precautions may result in exposure to the analyst and contamination of samples and the work environment, by airborne fibers. The cleanliness of the air in the work area is also ensured by testing the air quarterly with TEM analysis.

5.1   Sample Preparation

Gross examination of bulk samples is performed under low magnification (10-45X) to identify homogeneity, layering color, texture, friability and the presence or absence of fibrous constituents.

The sample is carefully removed from the sampling container and placed in an examination disk.   Sample integrity is maintained at this point in order to note any layering, and if possible, orientation of the top and bottom surfaces.   When discrete layers are identified, each is treated as a separate material, identifying and quantifying fibers in each layer. Each layer is analyzed and reported separately.

All fibrous materials are isolated (subsamples) and prepared for examination by polarized light microscopy. Isolation of these materials results in the loss of sample integrity since the sample must be "picked" through using forceps, probes, and needles. If the sample is not readily friable, a mortar and pestle can be used to crush the sample, or smooth jawed glazing pliers used to break the sample.

The type of sample matrix must be considered when determining sample preparation methodology. In samples such as floor tiles, roofing felts, tars, mastics and chalking, the fibrous materials of interest are often bound in a non-friable, organic substance, which makes observation of asbestos fibers difficult. Special techniques are used to reduce or remove these interference's such as ashing and solvent dissolution. These techniques are detailed below.

5.2   Sub-Sample Preparation

Representative sub-samples of suspect fibrous material must be obtained from a variety of matrix materials. In most cases, forceps and probes are sufficient to isolate fibrous materials for analysis by PLM.

Sub-samples are immersed in an appropriate refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope. A refractive index liquid is chosen based on the fiber's morphology as observed under the stereomicroscope.

The selection of appropriate procedures for identifying and collecting sub-samples is dependent on the sample matrix. The following are presented as sample preparation steps for typical bulk sample materials.

5.2.1   Spray-on Fireproofing and Acoustic Material

In general these materials contain some combination of cellulose, vermiculite, perlite, clay, binder, and possibly asbestos. They are very friable, and by nature of their preparation, are of a heterogeneous, mixed appearance. These materials are easily probed to isolate suspected fibrous materials.

The presence of cellulose, vermiculite and binder present their own unique problems

Cellulose may have approximately the same index of refraction as chrysotile-asbestos. For this reason, it is frequently confused with chrysotile. However, cellulose fibers frequently pinch and swell along their length exhibit internal cellular structure and lack splayed ends: they are not composed of bundles of smaller fibers.

Vermiculite may be confused with chrysotile. It has similar index of refraction and while it is not fibrous, its extinction characteristics under crossed polars may give the impression that the particles are composed of masses of matted fibers. The problem is compounded by the fact that chrysotile and vermiculite are a common mixture in sprayed-on coatings.

Sprayed-on binder materials may coat fibers and affect color or obscure optical characteristics. Fine particle of other materials may also adhere to fibers.

5.2.2 Cementitious Materials (plaster/transite)

These materials are usually non-friable, or not easily friable, and of a heterogeneous, mixed appearance, containing combinations of perlite, vermiculite, calcium carbonate, gypsum, hair, quartz, and possible wollastonite and/or asbestos.

Probing is not easy and a mortar and pestle may be necessary to facilitate size reduction of the sample.

This procedure is not recommended for samples, which contain amphibole minerals or vermiculite. Grinding of amphiboles may result in the separation of fiber bundles or the production of cleavage fragments, which have aspect ratios greater than 3:1 and will be classified as asbestos fibers. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

Transite board may be broken into pieces to expose "fresh" surfaces where fibers may be more easily identified.

Cleavage fragments of many natural minerals including amphiboles, talc, gypsum, wollastonite and vermiculite may appear as elongated anisotropic particles. The aspect ratio of these particles may be as great as 20:1. Therefore, aspect ratio alone is not sufficient for the identification of asbestos. Other properties of the asbestiform habit such as curved fibers, fiber bundles exhibiting splayed ends, and fibers with aspect ratios in excess of 20:1 must be observed in order to be sure asbestiform material is present in the sample. Therefore, once asbestos is known to be present, other properties (such as index of refraction an aspect ratio) can be used to identify asbestos and determine which particles will be counted in making a quantitative estimate of the amount of asbestos in the sample.

5.2.3 Thermal Insulating Material

These materials are usually very friable, white, and chalky in texture. Foam glass is an exception and may be covered by an outer layer (1/32 - 1/8") of asbestos-containing material. Careful attention must be given to identifying and investigating this layer. Suspected fibrous constituents can be easily isolated with probes and forceps. Problems associated with cellulose and vermiculite have been noted under the discussion about spray-on materials.

Certain minerals may be found in the construction materials, which are fibrous, or asbestiform, but which are not asbestos containing. These minerals include but are not limited to fibrous talc, fibrous brucite (nemalite), zeolites and dawsonite.

Fibrous glass including both mineral wool and fiberglass is very common in these materials. Its isotropic character makes it readily distinguishable from asbestos.

5.2.4 Ceiling Tiles

These materials are typically friable and have a fibrous matrix.

Three predominate mixtures of the constituents:

1)   cellulose, fibrous glass, perlite, filler/binder
2)   cellulose and filler/binder
3)   fibrous glass, filler/binder

When asbestos is present, it is usually either chrysotile or amosite. These materials are easily probed to isolate suspected fibrous materials. Particular attention should be given to the outer binder/paint layer of the three- (3) types mentioned. Type 3 is most likely to be asbestos containing.

5.2.5 Floor Coverings (Vinyl and Asphalt tiles, Linoleum)

Two types of materials are of concern in this category, block tile and sheet goods. They are non-friable (unless severely damaged by water, heat and weathering) and composed of a combination of tar, vinyl, quartz, calcium carbonate, and occasionally cellulose or fibrous glass. The mastic used to apply the product to the sub-floor is also a suspect for containing asbestos.

Isolation of suspect fibrous materials is not easily accomplished with probes and forceps. A "fresh" broken surface must be exposed to facilitate the location of any fibers. The use of the gravimetric reduction preparation methods is currently the best available for this sample type. See section 5.3

Caution must be exercised in handling these samples so that the mastic is not contaminated with debris and dust. Probe the mastic for potential fiber bundles. Clean as much of the mastic as possible off the fibers using a tungsten probe. A solvent such as acetone, chloroform, or the refractive index liquid can be used to wash off any remaining mastic.

Note: Dissolved mastic will change the refractive index of the RI liquid in which a fiber is mounted. If discoloration of the liquid is noted it may be "wicked" out from under the coverslip with a piece of paper towel. Fresh RI liquid can then be drawn, by capillary action, under the coverslip.

If fibrous material is not immediately apparent in the mastic, a smear of a small amount of mastic on a slide should be examined via PLM.

After the mastic is thoroughly examined, break or cut the tile to expose a "fresh" edge.

View these edges under the stereoscope to identify and isolate fibers or fiber bundles for PLM analysis.

The felt backing of vinyl sheet goods must be examined thoroughly. This material is easily probed for fibrous entities.

5.2.6 Asphalt Roofing Material

Roofing materials (roofing felts and asphalt shingles) are typically non-friable and have a heterogeneous, mixed matrix. Heavy probes must be used to tear through the material to isolate any fibrous materials. The use of the gravimetric reduction preparation methods is currently the best available for this sample type. See section 5.3 .

Solvent washing (acetone or RI liquid) may also be required to clean the fibers or bundles sufficiently for analysis. Fresh RI liquid must be used, after the cleaning, for PLM analysis.

5.2.7 Miscellaneous Materials (wall coverings, window and stage curtains)

Man-made fibers such as carbon, aluminum oxide, polyamides (nylon), polyester (Dacron) and polyolefins (polyethylene), and rayon are occasionally encountered in building materials.

The manufacturing process, and thus the physical form of these particles, can help to identify them. Typically, they are continuous, colorless, transparent cylinders with a round cross section. Two exceptions would be triacetate (Arnel and rayon fibers which demonstrate lengthwise striations and a multi-lobed cross section. Acrylic fibers show a cross section varying from kidney bean to dumbbell in shape. Synthetic fibers typically demonstrate high birefringence.

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5.2.8 Gravimetric Reduction

Using the combination of high temperature and solvent dissolution, a samples matrix may be removed (or reduced), facilitating the microscopic examination of the fiber of interest. Eliminating the interference's of tar, vinyl, calcium carbonate, etc. gives the analyst a clear view of the fibrous materials, allowing for the measurement of key optical properties.

Procedure:
Organic Reduction

- Weigh approximately .5-1.5 grams of the sample into a tarred crucible. Record weight.
- Ash sample at 480 degrees Celsius for a minimum of 6 hours
- Record weight of ashed sample
- Calculate the amount of organic constituent lost in %.

Inorganic Reduction

"Wash" remaining ash with .5 ml distilled water and add concentrated HCL. After 15 mins., dilute with additional distilled water. Pour into filtration apparatus with .4 micron polycarbonate filter. Apply vacuum. Dry filter in tarred plastic petri dish. Weigh filter and calculate % mineral lost.

5.3 Asbestos Identification

Positive identification of asbestos requires the determination of the following optical properties:

a.	morphology
b.	color and pleochroism
c.	refractive Indexes
d.	birefringence
e.	extinction characteristics
f.	sign of elongation

Table 5 lists the optical properties for a variety of fibrous constituents encountered in the analysis of building and insulation products. Table 7 presents a flow chart for the qualitative analysis of some of these materials. Central stop dispersion staining colors are listed in Table 6. It must be remembered that natural geological variations of asbestiform mineral deposits will produce exceptions to the data in Tables 5 and 6, and differences from laboratory standards.

The prepared slide is scanned identifying asbestos fibers using the optical properties of morphology, refractive Indices, color pleochroism, birefringence, extinction characteristics, sign of elongation and dispersion staining characteristics.

5.3.1 Pleochroism

This is a property exhibited by some colored anisotropic substances. When viewed by polarized light pleochroic crystals change color as they are rotated. Examine the fiber of interest in plane polarized light (i.e. polarized in, analyzer out), and observe any color changes which result as it is rotated through $360°$.

5.3.2 Isotropic/ Anisotropic

With the polarizer and analyzer crossed (i.e., dark field) rotate either the slide or the stage and observe the fiber of interest. An isotropic particle will remain dark (essentially invisible against the dark background). Conversely, anisotropic particles will present an image, which appears to fade in and out of the background (at $90°$ intervals) as it is rotated.

5.3.3. Angle of Extinction

As mentioned in Section 5.3.2., any anisotropic crystal extinguishes four times, between crossed polars, during a complete rotation.

This extinction occurs when the directions of vibration of the slow and fast rays of the fiber coincide with those of the polarizer and analyzer. Extinction may be one of three types:

1) Parallel or straight, when the fiber extinguishes parallel to the vibration direction the analyzer or polarizer (Figure I).
2) Symmetrical, when in the extinction position the vibration direction of the analyzer and polarizer are parallel to the diagnosis of a rhombic cross-section through a crystal (Figure I).
3) Oblique of inclined, when the fiber extinguishes at an oblique angle to the vibration directions of the analyzer and polarizer. This angle is known as the extinction angle, which is usually determined in terms of the slow vibration direction of the crystal.

FIGURE I. Types of Extinction

5.3.4 Birefringence

Birefringence (the difference between two indices of a particle on a given view) can be estimated from the interference colors observed when polarizers are crossed. As the stage (or slide) is rotated, isotropic particles (e.g., fibrous glass) will remain dark against the dark background. Particles with weak birefringence (e.g., quartz) will exhibit first order grays, whites, or yellows. As birefringence increases, higher order interference colors (reds, blues, greens, etc.) may be observed. As a rule, highly birefringent minerals appear brighter when rotated under crossed polarizers than do particles with weaker birefringence.

TABLE 1 - CATEGORIES OF BIREFRINGENCE STRENGTH WITH EXAMPLES

BIREFRINGENCE	INTERFERENCE COLOR IN SECTIONS 0.03 MM THICK	EXAMPLES, AND BIREFRINGENCE OF EXAMPLE
Weak: 0.0.010	First order gray, white or yellow	Apatite: 0.0003
Moderate: 0.010-0.025	First order red to second order green	Cancrinite: 0.0023-0.029
Strong: 0.025-0.100	Upper second order into fifth order	Zircon: 0.062
Very Strong: 0.100-0.200	High order-sixth and higher	Calcite: 0.172
Extreme: 0.200 and up	Very high order	Rutile: 0.285

5.3.5 Sign of Elongation

Using a first-order red 1 plate and crossed polars determine the sign of elongation by positioning the fiber at an angle of $45°$ to the analyzer and/or polarizer. When the slow ray of the red plate is parallel to the elongation of fiber, and the interference color of the fiber is yellow, the mineral has a negative sign of elongation. Vice versa, if the interference color of the fiber is blue, the mineral has positive sign of elongation. In other words, the arrangement of colors:

(in negative crystals)
yellow NW-SW elongation
blue SE-NW elongation

(in positive crystals)
yellow SE-NW elongation
blue NE-SW elongation
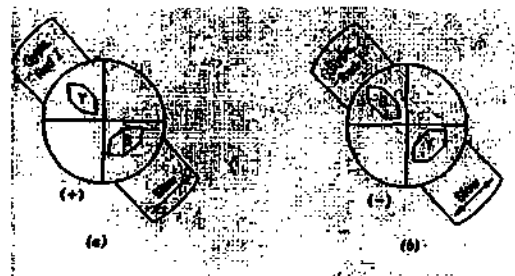
FIGURE II. Determination of Sign of Elongation

(a) positive elongation   (b) negative elongation

5.3.6 Dispersion Staining Colors

Dispersion staining is a technique for particle identification based on the difference between the dispersion of refractive index for a particle and the liquid medium in which the particle is immersed. In order to produce dispersion staining colors, the particle, and immersion liquid must have dispersion curves that intersect sharply in the visible light region. A special objective, containing annular and central stops in the back focal plane is required.

After isolating fibers of interest (sub-samples), follow the analysis flow chart (Table 7)

Note: Differences from standard characteristics may be observed due to natural variations in the conditions under which the minerals were formed and/or subjected to.

In the 1.55 HD refractive index oil, chrysotile will be readily identifiable from mineral wool or fiberglass by crossing the polars and using the 550-millimicron retardation plate to observe the colors of chrysotile. Both of the glass species are isotropic and will not show any colors. Many varieties of cellulose are close to 1.55 in index, but will not show chrysotile central dispersion staining colors. Characteristic magenta and blue colors identify chrysotile.

a.   If the fibers in the sample have a higher index of refraction than 1.55, have a negative sign of elongation, and appear blue by transmitted light, crocidolite is suspected. Prepare another slide with 1,700 refractive index oil. The color of crocidolite will be much bluer with an annular stop. The central stop dispersion staining colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If the fibers with the higher index than 1.55 are not blue, prepare a slide using 1.670 refractive index oil. Amosite has a positive sign of elongation and in the oil has central stop dispersion staining colors of yellow and magenta-blue.

b.   If the refractive index of the fibers is between 1.550 and 1.670 mount another preparation in 1.605 or 1.620 HD. The refractive indices for anthophyllite, tremolite, and actinolite vary naturally within the species. Anthophyllite can be distinguished from the other two by its parallel extinction. Actinolite has a light to dark green color with some pleochroism in transmitted light. The dispersion staining colors will have to be checked (i.e., actinolite DS colors in 1.63 RI oil are blue-magenta). A common interference mineral in this refractive index range is wollastonite. It also has a typical cleavage fragment morphology similar to the three asbestos minerals. Wollastonite has both a positive and a negative sign of elongation, parallel extinction and central stop dispersion stain colors in 1.605 HD of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is

needed, wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with a coverslip in place, on a warm hot plate until dry. By capillary action, place 1.62 refractive index oil under the slipcover and then examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show dispersion colors.

5.4   Quantification

If a sample is properly prepare (i.e., components are recognizable) it is possible to estimate its composition with considerable accuracy. It is desirable, though not always realistic, that the components be of the same size and specific gravity. Simple mixtures (2-3 components) can generally be analyzed to an accuracy of + 10%. Greater accuracy is attainable with practice. Comparison to a reference set of standard slides or photomicrographs can facilitate a more accurate analysis.

Always keep in mind that many conditions must be fulfilled in order to carry out a more or less accurate quantitative analysis. Three of the most important are:

1. The sample to be examined must be representative of the material to be analyzed. This will eliminate most of the errors arising from sampling.
2. The components of the sample must be distinguishable in appearance and/or properties. In some cases, additional treatment may be necessary to accentuate them.
3. Analysts ability to quantitative correctly as established with calibration and training.

After the asbestos species have been identified, scan the entire area under the coverslip of the slide with the 1.55 HD refractive index oil and determine an estimated volume percentage of each asbestos mineral. The slides with the other index oils can be used to help confirm these estimates, if needed.

1. Since complete homogeneity of the sample is often difficult to obtain, care must be exercised when estimating a single percentage.
2. If the estimate is in question, several microscopists can examine the slide and conclude amongst each other.
3. Estimate of the volume percentages of the other fibers in the sample can also be determined and reported as required

Make quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. Use either Calibrated Visual Estimation or Point Count technique.

Note: The point counting method only produces accurate quantitative data when the material on the slide has a uniform thickness, which is difficult to obtain. The point counting technique is recommended by the EPA and various State agencies to determine the amount of asbestos in bulk samples.

After performing qualitative analysis on each prepared slide, quantitative analysis must be performed by area estimation and recorded on the PLM worksheet.

Heterogeneous (Layered) Samples

For heterogeneous samples, a slide must be prepared for each distinct layer. A heterogeneous sample is a sample consisting of one or more analyses, which can be easily distinguished into layers and are not uniformly mixed. Each individual layer of a heterogeneous sample may have more than one material present.

From the prepared slide, examine a random field/graticle area at 100X under PLM. To determine the area percent of the materials visible, note the dispersion colors, the morphology and/or the sign of elongation for each different material. Estimate and record the percentage of each individual analyte to include asbestos fibers, non-asbestos fibers, and nonfibrous materials.

For each random field, the total area percentage must add up to 100%. A minimum of five random fields must be read from each slide preparation.

To determine the individual quantity of each analyte present in a homogeneous sample, find the average of the recorded area percentages for each. These percentages will be representative of the entire sample.

To determine the individual quantity of each analyte present in a heterogeneous sample the analyst must first determine the percentage of each distinct layer present in the sample as a whole. Examine the entire sample under the stereoscope to determine the layer percentages and record these percentages in decimal units. Find the average of the recorded area percentage for each individual analyte in each distinct layer. For each individual analyte in a layer, multiply the layer percentage (decimal) by the average area percent (whole number). When complete, add the percentages together to achieve a representative quantity of each analyte present for the entire sample. Use the PLM worksheet/layers form to enter data.

Calibrated Visual Estimation

The technique used for visual estimation is calibrated against the point count technique. To calibrate an analyst, he/she must quantitate a number of samples using the point count technique and reanalyze the same samples with visual estimation. The number of samples analyzed must be at a minimum of 20, in various ranges (see Monthly QC report). The data is compared using variance statistics as discussed below. This comparison of technique is performed until proficiency is established for all ranges.

Point Count

In addition to Calibrated Visual Estimation, the point count technique is a tool available to the analysts for the determination of asbestos concentration. Commonly used for petrographic applications, it is a widely accepted technique for the estimating asbestos concentrations based on a volume* percent of the binding matrix material. It can be used on a variety of material types, including friable matrices as well the residue ash of a non-friable organically bound material (NOB)

*Note: unless the densities of each of the materials being measured are known, concentrations based on weight percent are not achievable.

EPA Point-count method

This technique is outlined in EPA-600/M4-82-020, ' Interim Method for the Determination of Asbestos in Bulk Insulation Samples' and EPA/600/R-93/116, 'Method for the Determination of Asbestos in Bulk Building Materials'

Procedure:

1.   Prepare 8 slide mounts using the appropriate refractive index. Care should be taken to achieve a uniform distribution of materials throughout each slide prep.

2.   Using the single discrete point of a cross hair reticule, or Chatkey Point Array (a reticule with 25 "dots",) count points superimposed on either an asbestos or non asbestos matrix material for a total of 50 non empty points per slide preparation.

Reporting-
% asbestos is calculated:

% asbestos = (a/n) 100%
a = number of asbestos points
n = number of non asbestos points (total of 400-minimum)

If a=0, report ND
If $0 < a \leq 3$, report <1% asbestos

All values should be reported to the nearest %.

New York State, 198.1 (unofficially referred to as the "Stratified Point Count")

The State of New York has adopted the point count technique as the only acceptable quantitation method for friable building materials. NYS has incorporated a variety of modifications to the EPA's method

Procedure:
1. Prepare 4 slide mounts
2. For each slide preparation count all superimposed points until either **one** asbestos point is counted, or 50 nonempty points are counted.
3. If 4 asbestos points have been counted after all 4 preps have been analyzed, analysis can be halted.
4. If less than 4 points have been counted, 4 additional slides must be prepared and analyzed (at the rate of 50 nonempty points/prep) until: a) at least 4 asbestos points have been counted, or b) at least 400 nonempty points from the 8 slide preps have been counted.

Scanning Option: If the initial stereoscopic scan hints at the sample being negative, the analyst may opt to use the scanning option. All 4 mandatory slide preparations must be scanned at 100X mag. If no asbestos is detected the sample is reported as ND. The percentages of all other non-asbestos fibers may be determined by visual estimation. However, if asbestos is detected during this scan, the stratified point count must be initiated. The analyst begins the point count at the slide in which the asbestos was observed. Slides from that particular sample which were already scanned and contained no asbestos can be considered to contain 50 – non-asbestos points each.

Reporting

% asbestos = (a/n) 100%

a = number of asbestos points
n = number of non asbestos points (total of 400-minimum)

a = number of asbestos points
n = number of non asbestos points (total of 400-minimum)

Results are rounded off to 2 digits.

If no asbestos was detected, report ND
If the sample contained 0 (zero) asbestos points out of 400 (or more) non-empty points but did contain asbestos (observed during scanning) report <1%.

Note: this method does not refer to the "0 < a ≤ 3, report <1% asbestos" reporting protocol as indicated in the EPA method – see above.

Application:

The point count methods are used in a variety of applications. These include:
- samples analyzed under New York State regulatory guidelines
- as an additional quantitation tool to verify visual estimation values
- as a referee method for boarder line concentrations, <10%
- compliance with the NESHAP's ruling for sample concentrations <10%
- at the clients request
- for improved detection limits

Detection limit

Under normal conditions, the practical detection limit for this method is one (1) percent. Detection limits can vary with sample type, amount of sample analyzed or method of quantitation used. For example, the 1,000 point count method can report values down to 0.1%.

These detection limits are based on the limits as referenced in the documented methodology (EPA-600/M4-82-020, EPA/600/R-93/116). These limits have been widely accepted, recognized, and understood by the analytical community. A true specific method detection limit (MDL) study has not been performed by EMSL laboratories.

Results are reported for samples containing asbestos below the detection limits as "less than " one percent (<1%). With training in the use of known standards at various ranges of concentrations at, near and less than 1%, the analysts can accurately determine if the concentration of asbestos is <1% in the sample. Data collected from the comparison of point count data with visual estimation at levels below 1% is also a useful tool for the analysts. These procedures for 'the calibration of the analyst" is detailed in the EMSL training policies and procedures.

Any sample determined to contain no asbestos is to be reported as "none detected".

A note on trace: It is EMSL policy not to use the term 'trace' when reporting sample results, as this terminology is ill defined and ambiguous.

6.6   Blanks

Blank analysis is performed using NIST traceable materials (1866a).   Results are recorded daily to insure the absence of asbestos contamination in the work area. If contamination is found, its source must be immediately identified and eliminated.   Target sources include:

a.	Tools
b.	Slides
c.	Work areas
d.	Preparation agents (acids, solvents)
e.	Mounting oils

Non friable organically bound materials
At least one non- ACM NOB must be prepared and analyzed with every 20 samples. This includes the complete full preparation regime.

6.7   Daily Reference Slide Analysis

Samples are prepared from past proficiency testing samples with known concentrations and type of asbestos or NIST traceable standards. These samples are analyzed daily and recorded on applicable worksheets.   In addition to monitoring the accuracy of the analyst quantitation techniques, reference slide analysis is used to determine ability to measure optical properties.

Measurements are compared with the known standards.   The measurements recorded include:

- Refractive Index
- Birefringence
- Pleochroism & color
- Anisotropic/isotropic
- Sign of elongation
- Angle of extinction

The Laboratory Manager reviews the data for accuracy and records proficiency in the analysts files.

This data is entered into the monthly quality control report.   These results establish analyst accuracy as well as precision.

6.8 Proficiency Testing

All analysts in the NIST accredited PLM laboratories participate in the NIST's National Voluntary Laboratory Accreditation Program for Bulk Sample Analysis. Every active analyst must perform an individual analysis on the proficiency test sample, and report the data to the laboratory manager for review.

7.0   STATISTICAL ANALYSIS

Intra, Inter Analysts

The Quality Control calculations used for the Intra/Inter-analyst analysis based on a simple comparison of the relative difference of two values. These measures of variance are recorded and plotted over time to determine any trends or problems with the analysis.

Variance is calculated using:

For Intra-Analysts

$$R = |(A-B)|/((A+B)/2)|$$

Where: R = the measure of variance for the analysis

A = the value of the first analysis in %

B = the value of the second analysis in %

The Pass/Fail criteria for the QC analyses are as follows:

$R \leq 1$ - PASS

$R > 1$ - FAIL

Incorrect Asbestos ID - FAIL

Asbestos missed during analysis (false negative) - FAIL

Asbestos incorrectly identified to be present in a negative sample (false positive) - FAIL

For any failure of the above criteria the cause of the failure is identified and corrected. A deficiency /corrective action report is completed and place in the file.

For Inter-Analysts

$$R = (A-B)/((A+B)/2)$$

Where: R = the measure of variance for the analysis

A = the value of the first analysis in %

B = the value of the second analysis in %

The Pass/Fail criteria for the QC analyses are as follows:

$-1 \leq R \leq 1$ - PASS

$R > 1$ and $R < -1$ - FAIL

Incorrect Asbestos ID - FAIL

Asbestos missed during analysis (false negative) - FAIL

Asbestos incorrectly identified to be present in a negative sample (false positive) -FAIL

For any failure of the specified criteria the cause of the failure is identified and corrected. A deficiency /corrective action report is completed and place in the file.

For Standard Reference Analysis

Accuracy of analysis of standard, known samples, is determined using percent recovery calculations.   Results are quantified and charted to determine analyst, as well as laboratory precision and accuracy, using the following formula for Percent Recovery.

$$\%R = (A/F) \times 100$$

Where: %R = percent recovery

A = the analytical result
F = the formulated standard weight

Quality Control Data Management

The Management of QC data is reported, tracked, and analyzed using the EMSL Quality Control program. Using custom EXCEL spreadsheets, this program is designed to provide accuracy and precision information for all qualified analysts.

8.0 REFERENCES

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2. U.S. Environmental Protection Agency, "Bulk Sample Analysis for Asbestos Content;

3. Evaluation of the Tentative Method", EPA-600/4-82-021, May, 1982

4. McCrone, Walter, The Asbestos Particle Atlas, Ann Arbor Science, Michigan, 1980.

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7. NIOSH Manual of Analytical Methods, Method 7403-Asbestos Containing Materials in School Buildings: guidance Document, Parts 1 and 2, EPA/OTS No. C00090, March 1979.

8. Stoiber, Richard E. and Morse, Stearns A., Microscopic Identification of Crystals, The Ronald Press Company, New York,1972

9. EMSL.QAASB 101.0. Revision 1

TABLE 2 – ASBESTOS MINERALS, CHARACTERIZED BY THEIR MINERALOGY

Group	Designation of Mineral	Type of Asbestos	Chemical Abstracts Number[2]
Serpentine Amphibole	Serpentine	chrysotile	12001-29-5
	Riebeckite (glaucophane)	crocidolite(blue asbestos)	12001-28-4
	Grunerite (cummingtonite-grunerite)	grunerite asbestos (amosite)	12172-73-5
	Anthophyllite (gedrite)	anthophyllite asbestos[b]	77536-67-5
	Tremolite (ferroactinolite)	tremolite asbestos[b]	77536-68-6
	Tremolite - actinolite	actinolite asbestos	77536-66-4

TABLE 3 – CHEMICAL FORMULAS FOR TYPICAL ASBESTOS STRUCTURES

Mineral	Chemical Formula[c]
Chrysotile	$Mg_3 (si_2O_5)(OH)_4$
Amosite	$(Mg, Fe)_6(Si_8O_{22})(OH)_2$
Crocidolite	$Na_2Fe^{2+3} Fe^{3+2}(Si_8O_{22})OH)_2$
Anthophyllite	$Mg_7(Si_8O_{22})(OH)_2$
Cummingtonite	$(Mg, Fe)7(Si_8O_{22})(OH)_2$
Tremolite	$Ca_2Mg_5(Si_8O_{22})(OH)_2$
Ferroactinolite	$Ca_2Mg_5(Si_8O_{22})(OH)_2$
Actinolite	$Ca_2(Mg,Fe^{2+})_5(Si_8O_{22})(OH)_2$
Glaucophane	$Na, Mg_3, Al_2(Si_8O_{22})(OH)_2$

TABLE 4 - ASBESTOS AND ANALOG FORMS

Asbestos	Non-Asbestos Analog
Chrysotile	Angigorite-lizardite
Crocidolite	Riebeckite
Amosite	Cummingtonite-grunerite
Anthophyllite	Anthophyllite
Tremolite Asbestos	Tremolite
Actinolite Asbestos	Actinolite

a) Taken from ASTM Practice E849-82
b) These varieties have no special designations.
c) Taken from Dee, Howie and Zusman, Rock Forming Minerals, Vol. 3, Longmans, London, 1967.

TABLE 5 - OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology,Color[1]	Refractive Indices[2]		Birefringence	Extinction	Sign of Elongation
			α			
Chrysotile (asbestiform) serpentine	Wavy fibers. Fiber bodies have splayed ends and "kinks". Aspect ratio typically > 10:1. Colorless[4], pleochroic.	1.49-1.560	1.517-1.562[3] (normally 1.556)	.002-.014	\\ to fiber length	+ (length slow)
Amosite (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically > 10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present	1.635-1.696	1.655-1.729[3] (normally 1.696-1.710)	.010-.033	\\ to fiber length	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight, rigid fibers. Tick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color	1.654-1.701	1.668-1.717[3] (normally close to 1.700)	.014-.016	\\ to fiber length	- (length fast)
Anthophyllite	Straight, single fibers, some larger composite fibers. Anthophyllite cleavage fragments may be present with aspect ratios < 10:1[5]. Colorless to light brown	1.596-1.652	1.615 - 1.676[3]	.019-.024	\\ to fiber length	+ (length slow)
Tremolite-actinolite-asbestos	Tremolite-asbestos may be present as single or composite fibers. Tremolite cleavage fragments may be present as single crystals with aspect ratios< 10:1[5]. Colorless to pale green	1.599-1.668	1.622-1.688[3]	.023-.020	oblique extinction, 10-20° for fragments. Composite fibers show \\ extinction	+ (length slow)

1. From reference 7, colors cited are seen by observation with plane polarized light
2. From references 7 and 4
3. \\ to fiber length
4. Fibers subjected to heating may be brownish
5. Fibers defined as having aspect ratio > 3:1

TABLE 6 – CENTRAL STOP DISPERSION STAINING COLORS[a]

MINERAL	RI LIQUID	I	\\
Chrysotile	1.550[HD]	blue	Blue-magenta
"Amosite"	1.680	Blue-magenta to pale blue	Golden-yellow
	1.550[HD]	Yellow to White	Yellow to White
Crocidolite[b]	1.700	Red-magenta	Blue-magenta
	1.550[HD]	Yellow to White	Yellow to White
Anthophyllite-asbestos	1.605HD	Blue	Gold to gold-magenta
Tremolite-asbestos	1.605HDC	Pale-blue	Yellow
Actinolite-asbestos	1.605HD	Gold-magenta to blue	Gold
	1.630HDC	Magenta	Golden-yellow

a) From reference 3. Colors may vary slightly
b) Blue absorption color
c) Oblique extinction view

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TABLE 7:

POLARIZED LIGHT MICROSCOPY QUALITATIVE ANALYSIS: For each type of material identified by examination at low magnification. Mount specially dispersed sample in 1.550 RI liquid and view at 100x in plane polarized light and crossed polars.

Fibers Present

Fibers Absent

Analyze two additional preps at 100x

Fibers Present

Fibers Absent

Fibers are isotropic (disappear at all angles of stage rotation with crossed polars).

Fibers are anisotropic

Examination Complete

Possible fibers include:

Fiberglass	1-20 nm diameter
	RI typically <1.53
Mineral wool	0-200nm diameter
	RI typical <1.53
	non-uniform morphology

1. Determine extinction
2. Determine sign of elongation

SOE is Positive

SOE is Negative

n= 1.55
morphology = wavy (Chrysotile)
(Crocidolite)
morphology = ribbon (Cellulose)

n= 1.68
morphology = strait (Amosite)

n= 1.605
morphology = strait

n= 1.68
morphology = strait
pleochroism = blue

DS Colors = yellow - blue (Tremolite)
DS Colors = yellow - yellow (Actinolite)

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A.

MICROSCOPE CARE AND MAINTENANCE

GENERAL DISCUSSION

With careful treatment, most microscopes remain practically maintenance free for many years. General maintenance is confined to protection from dust, chemically aggressive substances and fumes, excessive temperatures (>45°C) and direct insulation. Specific precautions and instructions can be found in the manufacturer's operating manual(s).

EQUIPMENT

1.   Sable hair brush (clean and dry)

2.   Expanded polystyrene

3.   Cotton swabs

4.   Dust cover

5.   Rubber bulb blower or canned compressed air

6.   Chamois

REAGENTS

1.   Ether or non-chlorinated solvents such as xylene

2.   Distilled water

BACKGROUND & DEFINITIONS

See enclosures (FIGURES III, IV, IV, and TABLE 8)

METHOD

1. The optical parts of the microscope must be kept absolutely clean. All external surfaces and mechanical parts must also be cleaned periodically. Caution must be exercised during the use of solvents on any part of the microscope.

   Note: Alcohol and chlorinated solvents must never be used, which might destroy the cement between lens elements and the coatings on the exterior surfaces.

2. Dust on optical elements will degrade the image quality. Therefore, when not in use, the microscope must be covered with a dust cover. Special care should be taken to ensure that the tubes of the microscope are always closed with either an eyepiece or a dust plug.

3. When dust particles on the eyepiece will only give rise to patches in the image - which not every observer finds disturbing - a dirty objective front lens may hopelessly reduce the sharpness of the image, or at least its contrast. The eyepiece is close to the specimen, possibly to the immersion oil, but particularly to the hand operating the nosepiece. Due to these facts, the front lens of the objective is in special danger of becoming soil Even the lightest fingerprint may have grave consequences. Before starting important work, it is advisable - particularly if the microscope is not used by one observer alone. To unscrew every objective and check it carefully with the aid of a magnifier. Dirt is easily recognized if the objective is held so that the image of a light source is reflected from its plane surface. In the case of objectives that have a concave front lens, a different approach is indicated (to examine the surfaces of the front lens from the screw thread side). The remaining lens's elements can also be examined easily and any faults (cracks, "starting" of the cement) detected without difficulty.

4. The optical components of the polarized light microscope (PLM), arranged between the polars, are to be rotated from heavy mechanical stress in order to maintain the high degree of the optical isotropy of these components. This is to include things such as shock, fall, impact, tension, and pressure. For the same reason, the objectives of the microscope may be only slightly tightened against the contact face; stronger torsion would result in birefringence due to stress.

The cleaning of the objectives is confined to cleaning of the front and rear lenses with a dry brush and a rubber blower. Do not disassemble the objectives! Alcohol must not be used for cleaning purposes.

The knurled ring of the nosepiece should be turned, assuring that no objective is left in the light path during storage or transport of the microscope.

5. Should structures be found in the image which are suspected of being extraneous to the specimen, the fault may be traced as follows:

If the trouble can be eliminated by slight adjustment of the condenser, the cause must be sought in the bulb of the lamp, the lamp condenser, or the filter in front of it. However, if adjustment of the condenser does not produce results, the next step is to turn the focusing adjustment, which should eliminate all faults due to soiling of the condenser front lenses or the specimen. If this does not lead anywhere, slightly turn first the objective and then the eyepiece and you will immediately notice in which case the foreign body follows the rotation.

Dust particles are most clearly seen when the aperture diaphragm has been fully closed, because in this case the depth of focus is at its greatest.

6. In almost all cases, it will be sufficient to clean the outer lens faces with the aid of a grease-free brush. If necessary, wash first or with a frequently washed absolutely dust free linen rag (or chamois) and distilled water, (produced easily by breathing upon the surface to be cleaned).

If an organic solvent cannot be dispensed with, it is advisable to use very little ether or xylene instead of water, but never alcohol, which might destroy the cement between lens elements.

Ether is usually preferred because it evaporates most quickly and any harmful effect is thus less likely. Finally, residues are always removed with water as described above.

Should compressed air be available for cleaning, be sure to use a filter of cotton wool.

7. If the air in the workroom continuously has a relative humidity of more than 60%, certain precautions should be taken to avoid fungus growth on the optical elements. Do not keep microscopes under plastic covers. Do not store them in cabinets, but ensure good ventilation. If necessary, ventilate with the aid of a fan. In a particularly humid climate it is advisable to keep optical parts in perfectly air proof containers provided with a disinfectant or in which, for instance, a lamp and a fan circulate air of 40-50°C (100-120°F). The grease applied for corrosion protection on mechanical parts without surface coatings (operational surfaces) have to be renewed from time to time, the old grease to be removed by a solvent first.

8.   In case of electrical trouble, check first to see if the projection lamp is burnt out, or whether the fuse remains good.

     If the electrical trouble cannot be eliminated by replacement of the fuse or lamp, contact a repair representative.

9.   Repairs and maintenance, other than discussed in 5.1 thru 5.8 should not be attempted by any laboratory staff. Appropriate arrangements can be made with a local vendor who repairs and services microscopes.

.0   QUALITY CONTROL

1. An Instrument Record Card is maintained for each microscope. The information on this card is to include: model number, instrument type, serial number, I.D. number, location of instrument, checking/calibration frequency and responsibility, service representative and telephone number and a chronological record of all problems, repairs and calibrations.

2. An annual preventive maintenance visit is scheduled with a local service representative. This visit is beyond any service calls performed during the year.

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No. 30-g526-2.

7.3   F.K. Mollring, Microscopy from the Beginning, Carl Zeiss, Oberkochen, West Germany,
1968

7.4   W.J. Patzelt, Polarized-light microscopy, Ernst Leitz Wetzler GMBH, 1974.

7.5   Nikon Biological Microscopes, "Phase Contrast equipment Instructions:, Optiphot,
Labophot Phase Contrast Microscope, (84.8A) H&E-10S and (85.6B) H&E-3S.

TABLE 8: SUMMARY OF DAILY MAINTENANCE

External surfaces of objectives

Eyepieces, condensers:

Dust: remove with soft, dry sable brush.

Finger marks: remove immediately with a damp piece of linen or chamois leather; if necessary, use Light petrol.

Resistant dirt: remove with damp piece of fine linen or chamois leather.

Clean the lens first with a highly volatile solvent and allow all the solvent to evaporate.

Additional cleaning with expanded polystyrene has been found very reliable with dirt difficult to remove. The type of white, granular expanded polystyrene will known as packing material for instruments in particularly suitable. Break a piece off, press it against the dry lens with a protection grain of the fresh fracture surface, and rotate it as coaxially as possible with the lens axis. This removes, even from the recessed rims of the lens mount. Even the most minute residues of immersion oil, skin grease and solvents, which otherwise spread across the surface of the lens and partly counteract the reflection-reducing action of the coating layers. Any adherent grains of expanded polystyrene can be simply blown away or dislodged with an absolutely clean slate brush specially reserved for this purpose.

Cleaning is also possible with cotton wool wrapped around a wooden stick.

Oil immersion objectives:

Clean immediately after use: dab off with a piece of blotting paper or a small piece of linen. Remove the residual oil with a piece of linen soaked in light petrol.

Final cleaning: If necessary, with a petrol-soaked piece of linen. Never use methylated spirits or alcohol.

Internal surfaces of eyepieces

Condensers:

Dust: Blow it away softly or clean with a sable brush.

FIGURE III: STEREOSCOPIC ZOOM MICROSCOPE
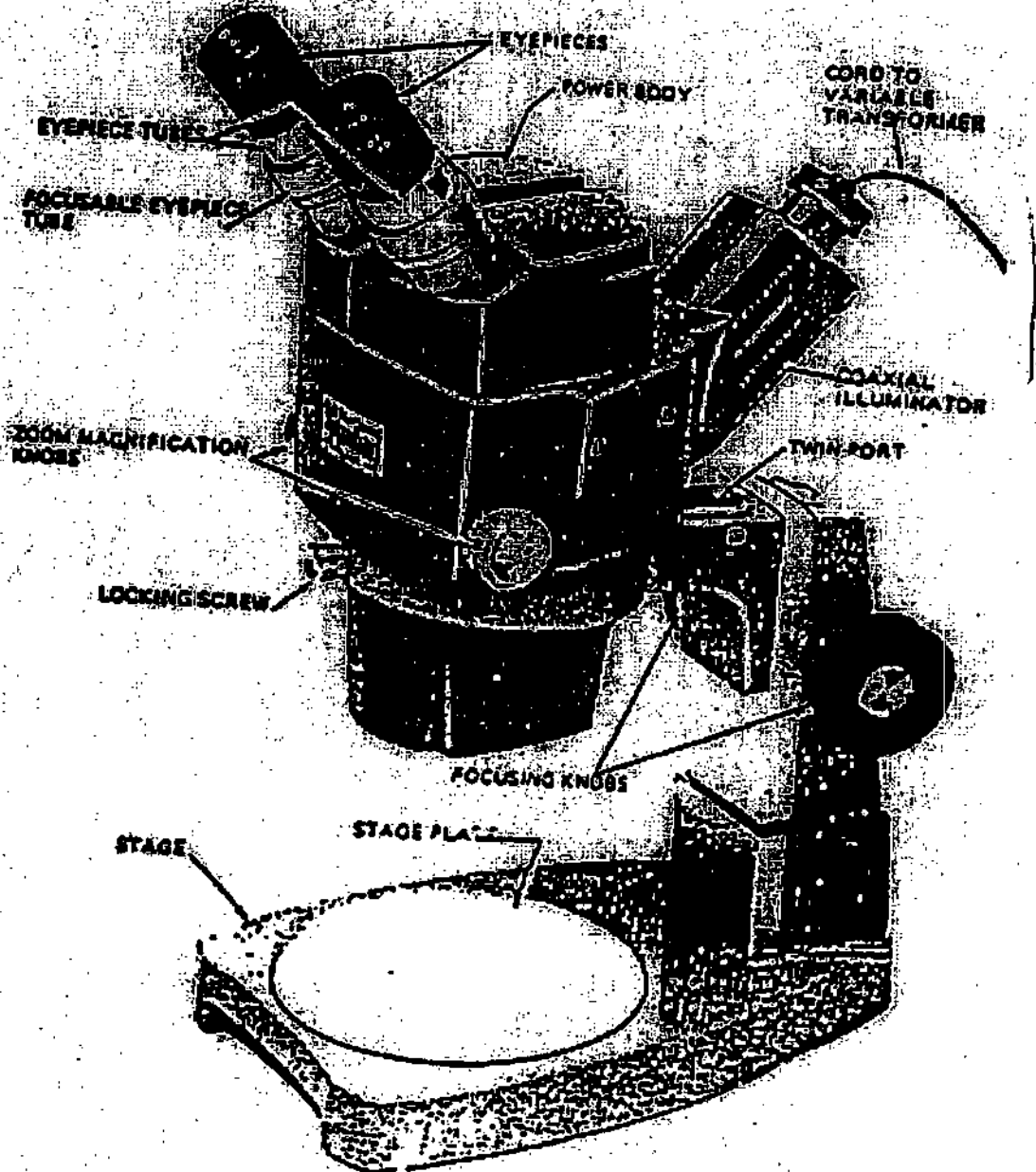
FIGURE IV: Polarizing Light Microscope
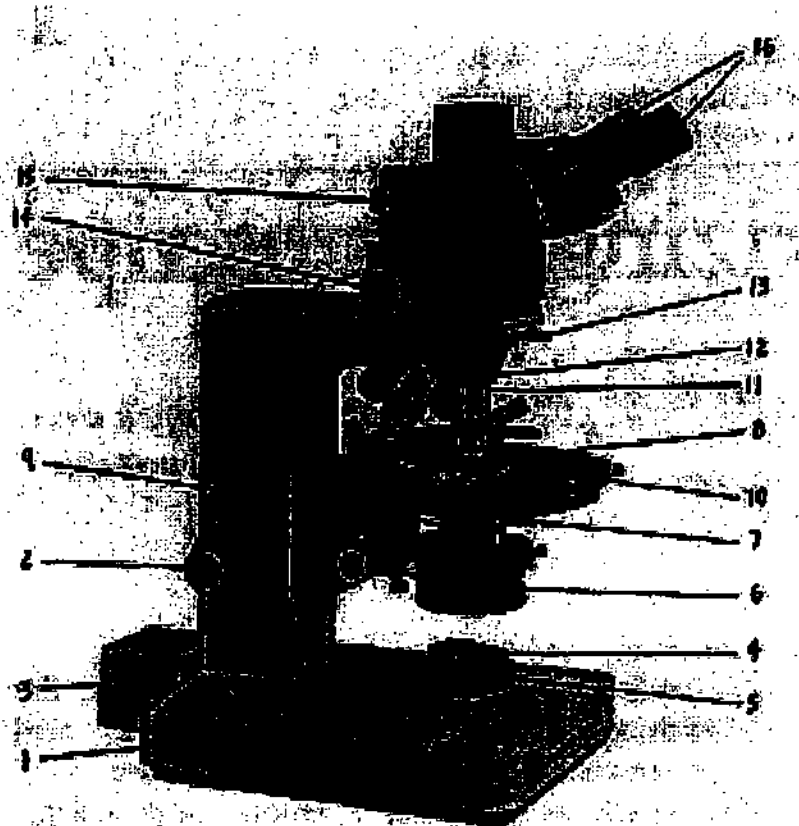
1.	Base	11.	Objective Lenses
2.	Course and Fine Focus	12.	Revolving Nose Piece for Objectives
3.	Illumination Source	13.	Analyzer
4.	Daylight Filter	14.	Bertrand Lens and Polerizer
5.	Field Iris Adjustment Control	15.	Camera Shutter Knob
6.	Rotatable Polarizer	16.	Oculars
7.	Substage Iris Diaphragm		
8.	Rotatable Stage		
9.	1.0 - 1.5x Condenser Lens		
10.	Slide Holder		

FIGURE V: POLARIZING LIGHT MICROSCOPE
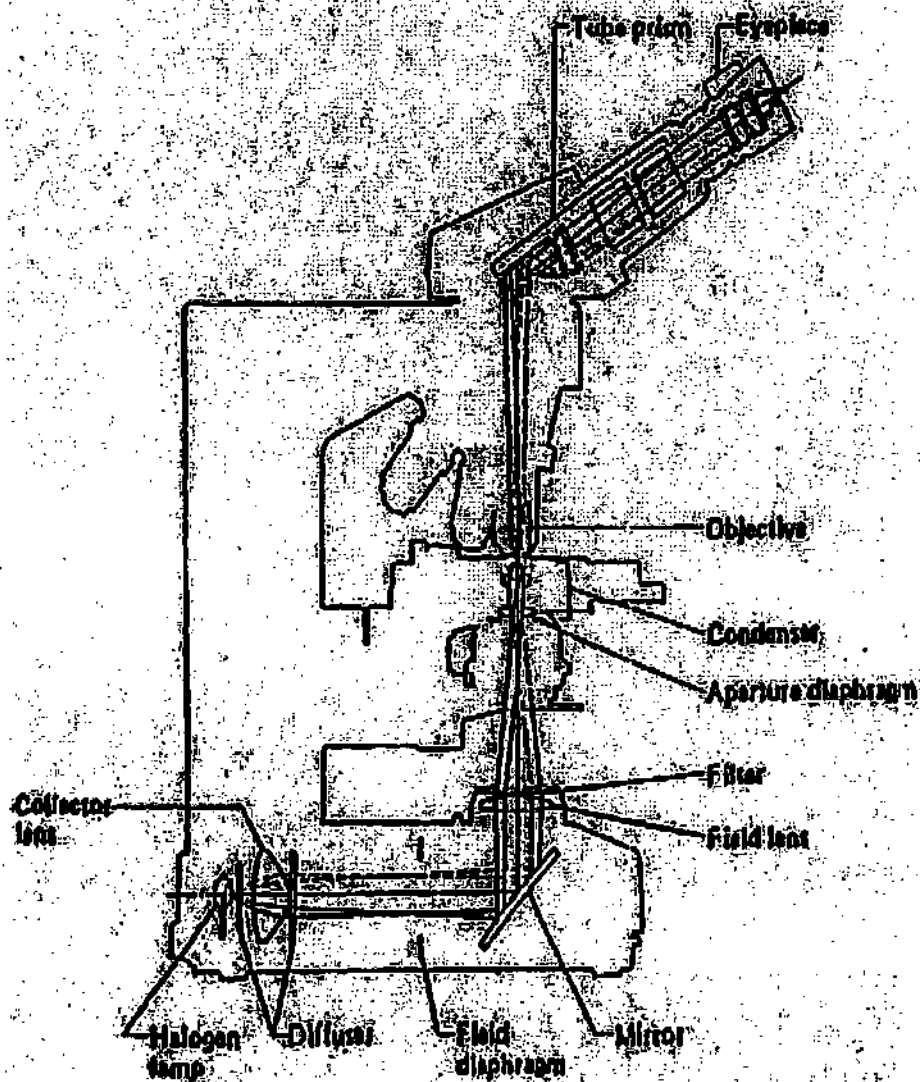
B.

ALIGNING THE OPTICAL SYSTEM

POLARIZED LIGHTMICROSCOPY (PLM)

1.0   GENERAL DISCUSSION

This procedure is to be used for aligning the optical system of the microscope for image formation of optimum quality, based on an illumination of the object with transmitted light.

2.0   EQUIPMENT

A compound microscope set up for polarized light microscopy to include a polarizer, analyzer, port for a wave retardation plate, a 360° graduated rotating stage, substage condenser, lamp and lamp iris.

Objective Lenses: 10X, 20-25X, 40-50X and a dispersion-staining objective.
Ocular lenses: 10X minimum.
Eyepiece Reticule: Cross hair.
Compensator Plate: 550-millimicron retardation (first-order red or gypsum).

3.0   BACKGROUND & DEFINITIONS

3.1   Polarizing Light Microscope (PLM)

This is an instrument for qualitative and quantitative work in either transmitted or reflected light. The majority of PLMs are used to study the optical properties of substances (e.g., geology, mineralogy, metallurgy, fiber research) in linearly polarized light.

The substage polarizer converts the unpolarized light of the light microscope into linearly polarized light. Between the objective and the eyepiece, there is a second polarizer, called the analyzer. Both are in calibrated mounts rotatable to 180° or 360°, and they can be swung out of the path of light. All optical elements between the polarizers must be strain-free. The objectives are individually centered. The tubes permit the use of an Amici-Bertrand lens to observe interference figures in the exit pupil of the objectives (conoscopic path of rays). The conoscopic image (e.g., together with a gypsum or quartz plate red first-order) renders information on the type of crystal examined (e.g. uni-axial or bi-axial, positive or negative). Most investigations require the polarizers crossed (directions of oscillation of polarizer analyzer oriented at 90° to each other), The crosshair of the oculars is congruent with these directions. The stage is of high precision and rotatable by 360°.

3.2 Light Source

The light source is generally an incandescent lamp with a concentrated filament for low voltage, in a pre-centered, pre-focused socket. The collator forms a magnified image of the light source in the plane of the aperture stop of the condenser, thus allowing for a light source of small dimension and low heat dissipation. The light intensity required for highest magnifications and binocular observations is much higher than required at lower magnifications. reductions of the light intensity can be made by interposing a neutral density filter. A less preferable alternate would be lowering the voltage to the lamp, with a resultant change of its "color temperature". This is not particularly objectionable for visual observations when a blue (daylight) filter is interposed in the light path, or for black and white photography, but must be considered when using color film. For polarized light microscopy a 100-watt incandescent lamp is preferred.

3.3 Condenser

The condenser, located in the substage, concentrates light on the specimen. The substage is generally permanently attached to the microscope. It should be equipped with a focusing device, for the condenser to focus the image of the field stop in the plane of the object. In addition, it should have a centering mount for the condenser, to center its optical axis with that of the objective (when the image of the field stop is concentric with the periphery of the field of view).

The numerical aperture of the condenser must be variable allowing for adjustment to that of the objective in order to obtain optimum resolution and contrast. The condenser images the field diaphragm into the specimen plane.

Selection of the components of the condenser system for illumination of the object from lowest to highest magnifications varies for different manufacturers. Optimum illumination conditions are explained in the manufacturer's instrument booklet.

3.4 Objectives

The microscope objective is the most important component of the optical system as it essentially determines the image quality. the majority of microscopes use two stage magnifications (objective plus ocular). The objectives required are divided into different categories according to their optical design and type of correction (e.g., achromats, fluorite, apochromats).

3.5 Oculars

The ocular at the upper end of the microscope tube enlarges the immediate image formed by the objective. Together with an attachment camera, it can also form the final image on a film plane. All oculars are derived from the either Huygens or Ramsden type.

Due to the low numerical aperture of oculars, only astigmatism, field curvature, distortion and the chromatic difference to magnification need to be corrected. The commonly used type of oculars is the compensating eyepiece, whose chromatic difference of magnification is equal but opposite to that of the objective. For use with flatfield objectives, differently corrected oculars are used in order to fully utilize the performance of the flatfield objectives. Many eyepieces have a high eye point to permit the microscopist to wear his corrective glasses.

Special eyepieces are available for measurements (filar eyepieces, micrometer eyepieces, interference eyepieces, image-splitting eyepieces, and so on) and for teaching purposes (pointer eyepieces and demonstration eyepieces). The magnifications range from 5X to 25X, of which 8X, 10X, 12.5X and 15X eyepieces are mainly used.

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4.0   METHOD

These instructions are specific for a binocular polarizing light microscope (PLM) equipped with the McCrone dispersion staining (DS) objective.

4.1   Alignment of the Illumination/Condenser System

Turn on the main switch and observe the light in the ground glass plate. If no light is observed, assure that the scope is plugged in, power is on and that the fuse and the bulb are in good condition. The illumination control should be increased to maximum. Assure uniform illumination in the object plane of the lower power objective.

Now an objective of medium magnification (10X) should be used so that when the field diaphragm is fully open, the entire field of view is illuminated. Focus the 10X objective and the 10X eyepiece on a specimen. The aperture diaphragm should be open and the field diaphragm closed just until its image is visible in the object field of the microscope (Figure VI). The condenser should be moved up and down until the image of the field diaphragm is as sharp as possible. The image will not be as sharp as that of the object, and color fringes may be visible at its edge (condenser too high the color is yellowish, too low and the color is bluish). When the field images is in best focus, center the image by means of the square socket wrenches.

With the image of the specimen and the centered field diaphragm in focus, the field diaphragm should be opened only so much that its image disappears at the edge of the field of view, but no further (Figure VI). When the diameter of the illuminated area is larger than the field of view, there is no increase in the intensity within this field, but the detrimental effect of glare increase.

FIGURE VI: OBJECTIVE DIAGRAMS
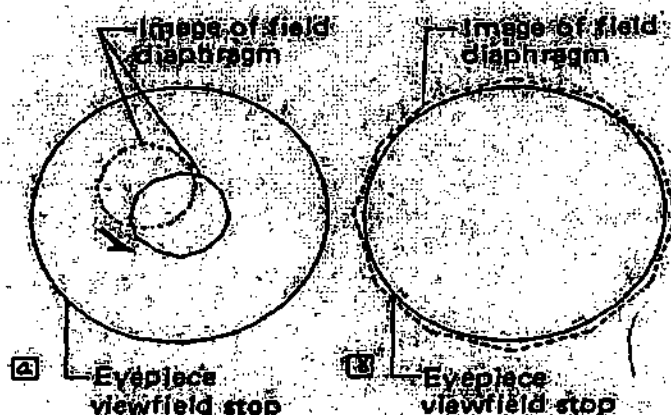
If the objective, required for a specific analysis, differs from that with which the alignment was made, it should now be placed in the light path and the alignment checked.

Note: Never swing an object into place by handling the objective directly. Always use the knurled ring of the nosepiece to rotate an objective into place. Unnecessary stress on the objective can create strain on the objective lenses, resulting in loss of image quality.

Insert the daylight filter into the filter holder and adjust lamp intensity according to individual needs.

The condenser should always remain in the positions as adjusted when objectives are changed, except for correction of concentration when necessary. Any displacement of the condenser in the direction of the optical axis (e.g., for varying the light intensity within the field of view), must be avoided. Many users of microscopes adhere to this incorrect procedure. Variations of the light intensity should be made by interposing a neutral density filter, or sometimes by varying the voltage of the light source (within certain limits and when variations in color balance are not important). When all the adjustments as described have been completed, and the aperture stop is still fully open, the image will appear with low contrast. The aperture stop should now be closed very gradually while the observer critically evaluates the image quality to detect slight improvements. You will notice that at a somewhat reduced numerical aperture, a fairly abrupt change in the optical quality of the image occurs. The contrast between the smallest resolved object detail increases slightly, the colors of stained objects appear slightly more "saturated"; there is a slight increase in the depth of field. At that position of the aperture stop, the image quality is at an optimum, and the microscope performs to the limit of its capacity with the selected optical components. This procedure is performed daily (see Calibration and Contamination Worksheet).

4.2   Centering the Objectives

Place a centering plate onto the specimen stage and focus it in the eyepiece with the aid of 10/0.25 achromatic objective. For this purpose, lift the stage carrier to the upper stop by actuating the combined control. Turning the control within the range of the two stops causes fine adjustment, turning the control beyond the stops causes coarse adjustment of the specimen. Bring the center of the centering cross and that of the eyepiece crosshair into coincidence, either by hand or with the aid of the objective traverser. Correct any deviations from this position occurring when the state is rotated, half by shifting specimen and half by turning the centering screws by means of the socket wrenches. Repeat this procedure until both crosshair centers remain in coincidence when the stage is rotated. According to the stage's center of rotation found in this way, adjust other objectives by means of centering screws so that the centers of the stage centering cross and eyepiece crosshair are in coincidence.

5.0   QUALITY CONTROL

1. An instrument Record Card is maintained for each microscope. Information on this card is to include: model number, instrument type, serial number, EMSL I.D. number, location of instrument, checking/calibration frequency and responsibility, service representative and telephone number and a chronological record of all problems repairs and calibrations.

2. An annual preventive maintenance visit is scheduled with a local representative. This visit is above and beyond any service calls performed during the year.

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6.2 Considine, D.M. (Editor), Van Nostrand's Scientific Encyclopedia, Volume II, Van Nostrand Reinhold Co., 1983

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C.

CALIBRATION OF REFRACTIVE INDEX LIQUIDS

1.0 OVERVIEW

Refractive index is one of the primary characteristics for the identification of asbestos. In order to accurately identify the RI of the particle it is important to know if the refractive index oil is within an acceptable range. (0.004) Two methods for the calibration of RI oils are documented here. The first is using an Abbe refractometer. The second is with Cargille glass standards and a method written by Dr. Shu-Chun Su.

The Refractometer:

1. Follow the manufacturers specifications for calibrating the instrument at 25°c using the water bath to stabilize the temperature.
2. After the instrument is calibrated, clean the prisms and stage.
3. Place 1 to 2 drops of RI oil onto the stage and allow to sit for one minute to stabilize the temperature of the oil.
4. Following the manufacturers instructions read and record the RI of the oil. It should be ± 0.004 the stated RI.

The Optical Glass Method: by Shu-Chun Su

2.0 MATERIALS & EQUIPMENT

1. Calibrated optical glass standards
2. Polarized Light Microscope with Dispersion Staining Objective
3. Thermometer with 1 degree divisions

3.0 METHODOLOGY

There are four steps in performing this calibration using optical glass standards and dispersion staining.

1. **Temperature**

   Find out what is the temperature, t, of the RI liquid to be calibrated. Generally, it is assumed that the temperature of the liquid is in equilibrium with the room temperature. If this is the case, the room temperature can be measured to represent the liquid temperature.

2. **Determine λ_o**

   Compare the liquid RI with that of an optical glass with accurately and precisely known RI's at various wavelengths to determine at which wavelength their RI values are equal. This wavelength is called the *matching wavelength*, λ_o, which can be derived from the dispersion staining color exhibited by the glass particles (See table 6).

3. **Determine n^L_D**

   Calculate n^L_D the RI value of the liquid at the wavelength of Fraunhofer spectral D line or 589nm and temperature t by using the following equation:

   $$n^L_D = n^S_D - (\Delta^L - \Delta^S) \cdot k_D$$

   Where: $n^L_D =$ the refractive index of the liquid at 589nm and t^oC
   $n^S_D =$ the RI value of the optical glass at 589nm, which is listed in the optical constant table supplied with every set of glasses

   $\Delta^L =$ The dispersion coefficient, $(n_F - n_c)$, of the liquid, which is printed on the bottle label

   $\Delta^S =$ The dispersion coefficient, $(n_F - n_c)$, of the liquid, which is listed in the optical constant table supplied with every set of glasses

   $k_D =$ a coefficient determined by the matching wavelength λ_o as listed in Table 6
   (Reference 3)

4. Determine n_D^{25deg}

If the room temperature is not 25°C, apply temperature correction to n'_D to find n_D^{25deg}, the RI value of the liquid at 25°C and 589nm. The equation used for temperature correction is;

$$n_D^{25} = n^t{}_D + (25-t) \cdot dn/dt,$$

Where: n_D^{25} = the RI of the liquid at 25°C and 589 nm
$n^t{}_D$ = the RI value of the liquid at 589 nm and $t°C$
t = the temperature in centigrade at which the calibration is performed
dn/dt = the temperature coefficient of the liquid, which is printed on the bottle label and is always a negative value for RI liquids.

No temperature correction is necessary for the glass if the temperature is within the range of 25° ± 10°C, because the temperature coefficient of Cargille glasses are so small that the resultant variation of RI will not exceed ± 0.0001.

PROCEDURE

1. Measure and record the room temperature with an accuracy of ± 2°C.
2. Select an optical glass standard whose RI is closest to that of the liquid to be calibrated, for example 1.55 glass for 1.550 liquid, 1.60 glass for 1.605 liquid.
3. Mount the glass in the liquid and observe the *predominant* dispersion staining (DS) color in central stop mode.
4. Convert the observed CS-DS color into the corresponding matching wavelength by referring to a DS color chart.
5. Convert λ_o and t into the corresponding n_D^{25} by referring to an appropriate conversion table:

Table 9: Refractive Index Oil Conversion Table

Cargille RI liquid Nominal or labeled n_D^{25}	Series	Cargille Optical Glass Standard Nominal or labeled RI	Lot No.	Conversion Table No.
1.550	E	1.55	B	3
			C	4
1.605	E	1.60	B	5
1.680	B	1.68	B	6
			C	7
1.700	B	1.70	B	8
			D	9

6. Compare the resulted n_D^{25} with the nominal or labeled value on the bottle of the RI liquid. If the absolute difference between the two values is less than or equal to 0.0044, this liquid can be used for bulk sample analysis. If the difference is greater than 0.004 then the liquid should be discarded.

7. Record the calibration result.

EXAMPLE:

If the Cargille 1.55 glass of Lot C is used to calibrate a Cargille 1.550 (Series E) RI Liquid at the room temperature of 21° and the predominant CS-DS color observed is bluish-purple, the corresponding λ_0 is then 570nm. $\lambda_0 = 570$ and $t = 21$ yield 1.548, which is the average of 1.5475 and 1.5485.

The calibration result shows that the RI of this bottle of 1.550 liquid at 589 nm and 25° is *actually* 1.548. Because the difference is 0.002, using $\pm$0.004 criterion, this RI liquid is considered acceptable for use in bulk sample analysis.

Charts are located in the appendix.

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5.0 QUALITY CONTROL

1. A Refractive Index Liquid calibration card (Figure VII) is maintained for each liquid. Information on this card is to include: manufacturer, catalog number, date received, received by whom, date RI verified, verified by whom, calibration responsibility and frequency, calibration instruction number, supplier, address and telephone number.

    The reverse side is to include: reference refractive index (n) and temperatures (T), measured n and room temperature (T_R), the corrected n, and the temperature coefficient.

2. A label containing the manufacturer's name, the initial calibration date, expiration date, and initials of the person performing the calibration will be attached to each bottle of RI liquid. The expiration date is 6 months from the time the oil was initially opened.

3. A RI liquid exhibiting a change in the refractive index of greater than = 0.005 must be replaced.

4. All RI liquids used daily (HD series 1.550, 1.605, 1.630, 1.680, 1.700) will be calibrated monthly.

5. The PLM QA/QC Manager at the main lab will receive copies of the data on a quarterly basis.

CALCULATIONS

As an example of the use of Equation 1, assume a mineral has been matched in index to a particular RI liquid (n_D = 1.530, T = 20°C, dn/dt = 3.00×10^{-4}), and the room temperature (TR) at the time of the match is 25° C. The actual refractive index of the liquid (and therefore the mineral) at the time of the match is:

$$n_{corr} = n_D + (T_R - T) \; dn \; dt$$
$$n_{corr} = 1.530 + (25 - 20)(-0.0003)$$
$$n_{corr} = 1.5285$$

The index reported for the mineral is then rounded off to 1.528. Reporting a refractive index of 1.5285 implies a greater accuracy than is attainable by this method (± 0.002).

7.0 REFERENCES

1.　　El-Hinnawi, Essam E., _Methods in Chemical and Mineral Microscopy_, Elsevier Publishing Co., 1966

2.　　Nesse, William D., _Introduction to Optical Mineralogy_, Oxform University Press, 1986

3.　　Bloss, F. Donald, _An Introduction to the Methods of Optical Crysallography_. Holt, Rinehart Winston, 1972

4.　　Schaeffer, Harold F., _Microscopy for Chemists_, Dover Publications, Inc. 1953

5.　　Air pollution Training Institute, "Course 420 - Air Pollution Microscopy Laboratory Manual", June 1979

FIGURE VII - REFRACTIVE INDEX LIQUID CALIBRATION CARD

Manufacturer: _____   Date Recv'd: _____

Catalog #: _____   Recv'd by: _____

Batch #: _____   Date R.I. verified: _____

Assigned to: _____   Verified by: _____

Calibration Responsibility: _____

Calibration Instruction #: _____

Supplier: _____

Telephone #: _____   Address: _____

_____

Comments: _____

	Reference		Measured		Corrected	Calibration		Accept/
n_D	T	dn/dt	n	T	n	Date	Initials	Reject

TABLE 10: THE PROBABLE ERROR IN REFRACTIVE INDEX DETERMINATION

METHOD USED	PROBABLE ERROR (±)
Central illumination	0.002 - 0.003
Becke line colors	0.002
Oblique illumination	
Single diaphragm	0.002 - 0.003
Double diaphragm	0.0001 - 0.0002
Dark-field immersion method	0.001 - 0.002
Focal screening method	
Apertural screening	0.001 - 0.002
Unilateral screening	0.0001 - 0.002
Central screening	0.001
Variation methods	
Thermal variation	0.001
Dispersion	0.001
Double variation	0.0004
Glass method	0.0002 - 0.0003

FIGURE VIII: BECKE LINE

A.   GRAIN INDEX IS GREATER THEN OIL'S
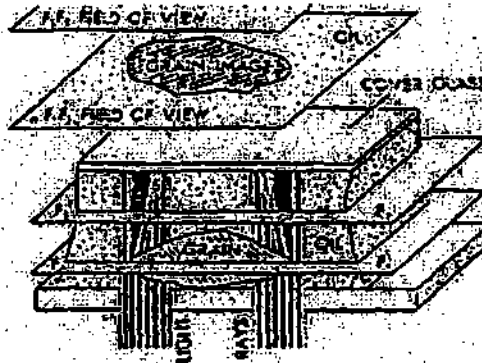
B.   GRAIN THAN OIL'S   INDEX IS LESS
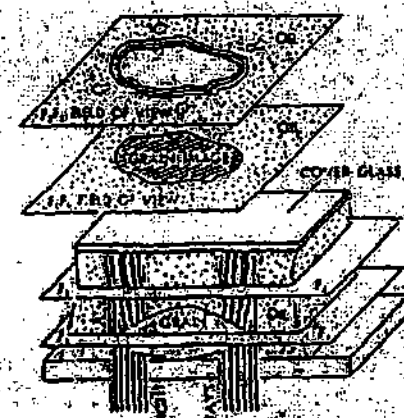
The disposition of movement of Becke Lines, field of view, F_1F_1 is that observed if the microscope is focused upon plan F_1F_1 to produce the sharpest image; here the Becke Lines are not too obvious. However, if observed while the microscope is racked upward toward a focus on plan F_2F_2, the Becke Lines become increasingly apparent, the brighter lines moving (as indicated by the hollow arrows) toward the medium having the greater refractive index.
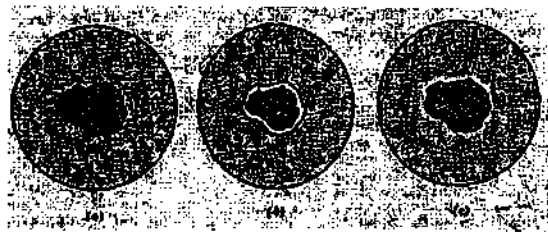
a) oil = particle: b) oil < particle: c) oil > particle

FIGURE IX: BECKE LINE MOVEMENT
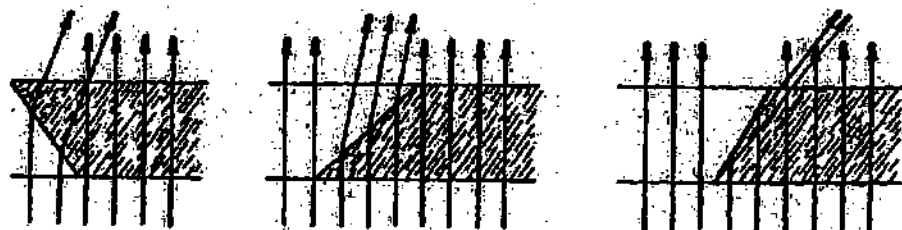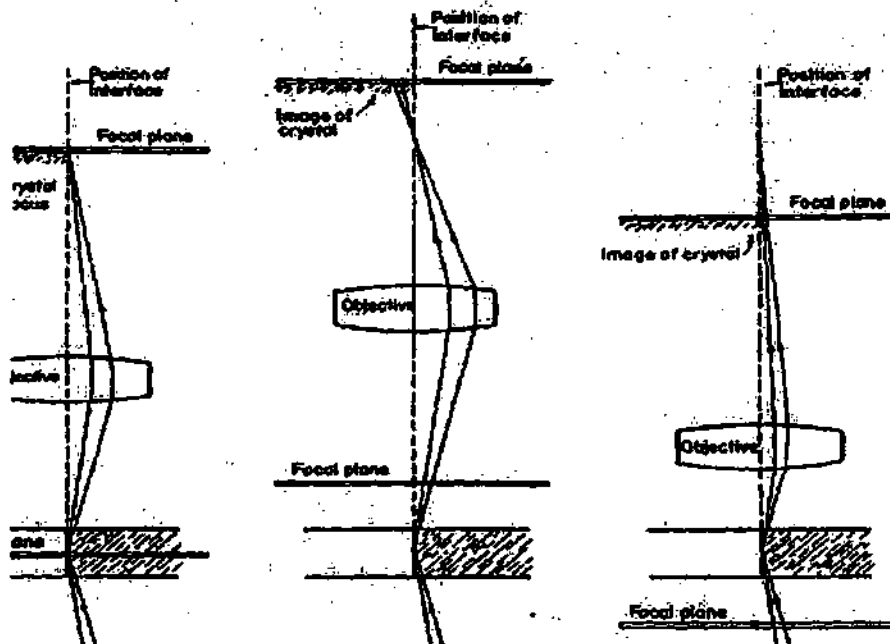
D.

ASBESTOS & NONASBESTOS MINERAL DESCRIPTIVES

Mineral Information

Asbestos:		*Non-Asbestos:*	
	Chrysotile		Augite
	Anthophyllite		Diopside
	Actinolite		Hedenbergite
	Tremolite		Enstatite
	Amosite		Hyperstene
	Crocidolite		Halloysite
			Kaolinite
			Palygorskite
			Talc
			Wollastonite

ASBESTOS MINERALS

ANTIGORITE, LIZARDITE, & CHRYSOTILE

Class: Phyllosilicate
Group: Serpentine

Antigorite	$Mg_3Si_2O_5(OH)_4$
Chrysotile	$Mg_3Si_2O_5(OH)_4$

Color: various shades of green; brownish, gray, white, or yellow: translucent to opaque.
Luster: waxy or greasy; fibrous varieties silky; massive varieties earthy.
Transparency: translucent to opaque
Habit: Occurs mainly as fibrous chrysotile or as lamellar or platy antigorite.
Antigorite & Lizardite: Massive, fine grained, lamellar
Chrysotile: Fibrous
Hardness / Specific Gravity: variable 2.5 -5.0 / 2.5 - 2.6
Cleavage / Fracture: Basal, perfect / conical, splintery

Antigorite

Optics: (+)

Pleochroism = none
Refractive Indices: 1.55-1.56
Birefringence: low (0.007)

Extinction Angle: 0, 90°

Crystallography: Monoclinic; 2/m (Orthorhombic Antigorite & Chrysotile are rare)
a=5.30; b=9.20; c=7.46; B=91°24' a:b:c=0.567:1:0.811
Z= 2. d's: 7.30(10), 3.63(8), 2.52(2), 2.42(2), 2.19(1)
Composition: MgO 43, SiO2 44.1, H2O 12.9
Ni & Fe may sub. for Mg and Al for Si
Cleavage: {001} basal, perfect. Corrugated, finite layers parallel to {001}

Chrysotile

Optics: (+)

Pleochroism: none
Refractive Indices: x= 1.55-1.56   y= 1.545-1.556
Birefringence: Moderate (0.011)

Extinction angle: 0- 90°

Crystallography: monoclinic; 2/ m (Orthorhombic Antigorite & Chrysotile are rare)
a=5.34 b=9.25 c=14.65 β=93°16' a:b:c=0.577:1:1.584
Z= 4.
Composition: MgO 43, SiO2 44.1, H2O 12.9%
Ni & Fe may sub. for Mg and Al for Si
K - Factor Ratios: Na=0.00, Mg=0.70, Si=1.0, Ca=0.00, Fe<0.02
Cleavage: none. A mismatch between the t and o layers causes the structure to scroll forming cylindrical tubes

Distinguishing features:
color, luster, smooth, greasy feel.

Optically:

(chrysotile asbestos) wavy bundles under polarized light and scanning electron microscope; hollow, tubular fibrils in transmission electron microscope. Antigorite; plate aggregates under PLM & SEM; Plate aggregates with perfect cleavage under TEM.

Occurrence:

Serpentines are secondary minerals formed from other deposits such as olivine; orthopyroxene, amphibole, and magnesium silicates. They are found in both igneous and metamorphic rock formations.

ANTHOPHYLLITE

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Class: Inosilicate
Group: Amphibole

Anthophyllite   (Mg, Fe)7 Si8 O22 (OH)2

Color: white, gray green, & brown. (brown predominates in cummingtonite series.)
Luster: vitreous, silky for fibrous varieties.
Transparency: Translucent. Transmits light on thin edges.
Twinning: Common.
Habit::   Orthorhombic; 2/m 2/m 2/m.   Distinct crystals are rare. Commonly lamellar, fibrous,
    radiating, slender & prismatic or aggregates of fibrous crystals.
Hardness / Specific Gravity: 5.0 - 6.0 / 2.8 - 3.4
Cleavage / Fracture: {210} prismatic, perfect.

Optics: (+)
    Pleochroism : none
    α=1.60-1.69, β=1.61-1.71, γ=1.62-1.72. Indices will increase with Fe.
    2V=70-100°, X=a, Y=b. absorption Z > Y and X.
    Birefringence: moderate (0.02)
Pleochroism: none

Crystallography:

Pnma; a=18.56;   b=18.06;   c=5.28; β= 90°0'   a:b:c=1.027:1:0.292
Z= 4. d's: 8.26(6), 3.65(4), 3.24(6), 3.05(10), 2.84(4)
(210)^(2-10) = 55°

Composition:

Forms a solid solution series from Mg7Si8O22(OH)2 to Fe2Mg5Si8O22(OH)2
At higher Fe concentrations, cummingtonite results.

Distinguishing Features:

Light brown color. Needle like or fibrous, often radiating habit. Indistinguishable from cummingtonite-grunerite series with out microanalysis. Lack of reaction to HCl. Thin plate like fibers under TEM. Anthophyllite can form In the same environment as talc and during elemental analysis may contain similar Mg to Si ratios. In this scenario, the diffraction pattern is the key to distinguishing between the two structures. Talc has a hexagonal pattern while anthophyllite primarily yields a series of parallel spots clearly marking the (h,k,0) and the (h,k,l) rows. In addition, the c axis may contain alternate or (missing) spots as in the [010], [140] & [320] zone axis. Fusible at 5 to a magnetic black enamel.

CUMMINGTONITE
GRUNERITE (Amosite Asbestos)

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Class: Inosilicate
Group: Amphibole

Cummingtonite	$(Mg, Fe)_7 Si_8 O_{22} (OH)_2$
Grunerite	$Fe_7 Si_8 O_{22} (OH)_2$

Color: White-gray. Varying shades of lt. brown are common in cummingtonite series.
Transparency: Translucent. Transmits light on thin edges.
Luster: Vitreous. Silky in fibrous form.
Twinning: Common.
Habit:: Monoclinic; (Cummingtonite 2/m; Grunerite C2/m). Distinct crystals are rare. Commonly Lamellar, fibrous, radiating, slender & prismatic or aggregates of fibrous crystals.
Hardness / Specific Gravity: 5.0 - 6.0 / 2.8 - 3.6
Cleavage / Fracture: {110} prismatic, perfect.

Optics: (-) grunerite; (+) cummingtonite; (heated grunerite as amosite)
α= 1.65-1.69 (-1.74); β= 1.67-1.71 (-1.95); γ= 1.69-1.73

2V large; Y = b; Z ^ c= 13-20°; r<v for cummingtonite; r>v for grunerite.
Pleochroism: none (May appear yellow to red)
Birefringence: Moderate 0.03 (up to 0.19)

Crystallography: Monoclinic
a= 9.59, b= 18.44, c= 5.34, β= 102°0'. Unit cell length decreases with increase in Mg.
a:b:c= 0.520:1:0.289
Z= 2. d's; 9.21(5), 8.33(10), 3.07(8), 2.76(9), 2.51(6)

Composition:
A solid solution series exists between cummingtonite and grunerite starting from approximately $Fe_2 Mg_5 Si_8 O_{22} (OH)_2$ to $Fe_7 Si_8 O_{22} (OH)_2$. Members with an atomic percentage of Mg > Fe is referred to as cummingtonite. 30 atomic percent is used as a division between members. Al2O may range up to 0.4 weight percent and CaO as high as 0.9 percent.

Distinguishing Features:
Light brown color. Needle like or fibrous, often radiating habit. Indistinguishable from anthophyllite with the naked eye. Lack of reaction to HCl. Fe up to 80% that of Si. Thin plate like fibers under TEM with distinctive, easily obtained diffraction patterns.

GLAUCOPHANE
RIEBECKITE (Crocidolite Asbestos)

Class: Phyllosilicate
Group: Amphibole

Glaucophane   Na2 Mg3 Al2 Si8 O22 (OH)2
Riebeckite   Na2 Fe3^{2+} Fe2^{3+} Si8 O22 (OH)2

Color: Glaucophane is blue-grey, to lavender-blue. Riebeckite is dark blue to blue-black. Darker with increased Fe content.
Transparency: Translucent
Luster: Vitreous, silky in fibrous varieties.
Habit: Monoclinic; 2/m   Slender acicular crystals, aggregated, or in fibrous form.(Crocidolite Asbestos)
Hardness / Specific Gravity: 5.5 - 6.0 / 3.0 - 3.4
Cleavage / Fracture: {110} prismatic, good / Uneven.

Optics: (-)
Pleochroism = blue to blue-grey. X<Y<Z
Refractive Indices: α=1.61-1.70, β=1.62-1.71, γ=1.63-1.72
Birefringence: moderate (0.045)

Extinction Angle: 0, 90°
Glaucophane, (Riebeckite)

Crystallography:
C2/m; a=9.58 (9.769); b=17.80 (18.048); c=5.30 (5.335);
a=90°, β=103°48' (103°59'); a:b:c = 0.538:1:0.298
Z= 2. d's: 8.42(10), 4.52(5), 3.43(6), 3.09(8), 2.72(10).
2V = 40 - 90°; Y = b, Z^c = 8°; X < Y < Z.

Composition:

The composition changes with the substitution of Fe^{2+} for Mg and Fe^{3+} for Al.
Care should be taken during chemical analysis as the elemental make up of Riebeckite may closely resemble that of Grunerite. The distinguishing feature that is most prominent is the occurrence of Na. Also known as crocidolite in its fibrous form.

Occurrence:

Glaucophane is found in metamorphic rocks such as marble and schists. Reibeckite most commonly occurs in igneous rocks. Tiger-eye is an ornamental stone where quartz has taken the place of crocidolite while preserving a fibrous texture.

ACTINOLITE
TREMOLITE

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Class: Inosilicate
Group: Amphibole

Actinolite   Ca2 (Mg, Fe)5 Si8 O22 (OH)2
Tremolite   Ca2 Mg5 Si8 O22 (OH)2

Color: white to gray (tremolite), light to dark green (actinolite)
Luster: vitreous, often with silky sheen.
Transparency: transparent to translucent
Twinning: common
Habit::   usually aggregates of fibrous crystals. Individual crystals are rare.
Hardness / Specific Gravity: 5-6 / 3.0 - 3.3
Cleavage / Fracture: {110} perfect

Optics:

	SHAPE	PLEOCHROMISM	INDICES	BIREFRINGENCE	EXTINCTION
TREMOLITE	fibrous	none	1.60 - 1.62	mod. 0.02	0 - 5
	non-fibrous	none	1.60 - 1.62	mod. 0.02	0 - 20
ACTINOLITE	fibrous	none	1.63 - 1.65	mod. 0.02	0 - 5
	non-fibrous	none	1.63 - 1.65	mod. 0.02	0 - 16

Crystallography: Monoclinic; 2/m
Actinolite, (Tremolite)
C2/m; a=9.86 (9.84); b=18.11 (18.02); c=5.34 (5.27);
a=90° β=105° (104°95');   a:b:c= 0.545:1:0.293
Z= 2. d's: 8.38(10), 3.27(8), 3.12(10), 2.81(5), 2.71(9)
Orientation: 2V = 80°; Y = b, Z^c = 15°

Composition:

There is a solid solution series from tremolite to ferroactinolite.
(Ca2Fe5Mg5Si8O22(OH)2.)

Distinguishing features:

Radiating tremolite may resemble wollastonite however can be distinguished by a lack of
reaction with HCl. Actinolite is lighter in color than the majority of hornblende's.

Optically

Thin strait fibers under PLM & SEM analysis with a slender prismatic habit being evident.
TEM analysis reveals platy bundles with good cleavage giving the edges a choppy look.
The chemical composition of the structure is a good indication of its nature.

Occurrence:

Tremolite commonly occurs in metamorphosed dolomitic limestone's and may, in rare instances, form in some serpentines. Radiating tremolite may resemble wollastonite, however it shows no reaction to HCl that is a distinguishing feature of wollastonite. A felted aggregate of tremolite fibers is also known as *mountain leather or mountain cork.*

Actinolite usually forms in green schist's produced from low to medium grade metamorphism. It also coexists with quartz and epidote. A tough, compact variety that supplies much of the material for *jade* is called *nephrite (jadeite).*

NONASBESTOS MINERALS

AUGITE
DIOPSIDE
HEDENBERGITE

Class: Inosilicate
Group: Pyroxene (Clinopyroxene)

Augite $(Ca,Na)(Mg,Fe,Al)(Si,Al)2O6$
Diopside $CaMgSi2O6$
Hedenbergite $CaFeSi2O6$

Color: White to pale green & black. Darkness increasing with Fe towards the black of augite.
Luster: vitreous
Transparency: transparent, translucent to opaque
Twinning: common
Habit: crystals usually square or eight-sided cross section; also massive, granular, lamellar
Hardness / Specific Gravity: 5.5 - 6.5 / 3.2 - 3.6

Cleavage / Fracture: {110}, at 87° & 93° imperfect. Frequently parting on {001} and less commonly on {100}. Fracture is uneven.

Optics: (+)
    Pleochroism : varies in darker members only.
    (X=pale green, Y=Yellow-green, Z=dark green)
    Refractive Indices: α=1.66-1.73, β=1.67-1.73, γ=1.69-1.75
    2V=55-65° , Y=b, Z^c=39-48°; r>v.

Crystallography: Monoclinic; .2/m

    C2/c;   a=9.73;   b=8.91;   c=5.25; β=105°50';   a:b:c=1.092:1:0.589
    Augite   a=9.755;   b=8.928;   c=5.204; β=106°11'
    Diopside   a=9.761;   b=8.926;   c=5.258; β=105°79'
    Hedenburgite   a=9.827;   b=8.994;   c=5.261; β=105°52'
    Z= 4. d's: 3.23(8),   2.98(10),   2.94(7),   2.53(4),   1.748(4).
    m(110)^m'(1-10)=92°50',   c(001)^(p(111)=33°50',
    s'(-111)^s'(-1-11)=59°11',   c(001)^a(100)=74°10'.

Composition;
    solid solution series exists between all members of this group toward acmite-augite. A solid solution series exists between diopside and hedenbergite with Mg and Fe^{2+} substitution. Most of the members of this group have a 1 - 3% Al_2O_3 content. Augite has substitution in Mg - Fe^{2+} and Al substitutes for Mg, Fe^{2+} & Si. Na and Ti may also be present.

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Distinguishing Features:

Two cleavages almost at right angles (87 & 93°). Augite is usually a darker green than diopside. Insoluble in acids. Fusible at 4 to a green glass.

Occurrence:

Diopside and hedenbergite are native to metamorphic rock formations. Diopside occurs in impure limestone and occasionally in basaltic igneous rocks.

ENSTATITE
HYPERSTHENE

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Class: Inosilicate
Group: Pyroxene

Enstatite $MgSiO_3$
Hyperstene $(Mg, Fe)SiO_3$

Color: Pale green to dark brownish green, yellowish, or greenish-white.
Luster: vitreous, pearly on cleavage surfaces. Translucent to opaque.
Transparency: Translucent to opaque
Habit: Crystals are prismatic; usually grains or massive; fibrous , lamellar
Hardness / Specific Gravity: 5.0 - 6.0   /   3.2 - 4.0
Cleavage / Fracture: Prismatic, good {210}, {100} & {001} are less common. Fracture is uneven.

Optics: enstatite (+), Hyperstene (-)
Pleochroism : varies
Refractive indices: α=1.650-1.715, β=1.653-1.728, γ=1.658-1.731
2V=35-50°, X=b, Z=c. Indices increase with Fe.

Crystallography: Orthorhombic; 2/m 2/m 2/m
Pbca; a=18.22;   b=8.81;   c=5.21;   β= 90.00 a:b:c=2.068:1:0.590
Z= 8. d's: 3.17(10), 2.94(4), 2.87(9), 2.53(4), 2.49(5)
(210)^(2-10) = 91°44'

Composition:

Fe:Mg ratios rarely exceed 1:1. Pure enstatite contains no Fe. Mg may be substituted for by Fe^{2+} up to 90%.

Distinguishing features:

Two cleavages may appear to intersect at or near right angles. Varieties that are high in Fe content may appear almost black, similar to augite in appearance. Enstatite-Hyperstene is commonly recognized by its cleavage and luster. Thin, nearly infusible, edge may be rounded. Fibers are common under high magnification. It is commonly located in metamorphic rock and may coexist with amphiboles.

Occurrence:

Igneous rock such as peroxenite. They may give way to amphiboles especially anthophyllite in metamorphic rocks.

HALLOYSITE

Class: Phyllosilicate
Group: Clay Mineral

Halloysite Al2 Si_2 O_5 (OH)$_4$ & Al_2 Si_2 O_5 (OH)$_4$ • $2H_2O$ *dehydrates to the first composition with
the loss of inner layer water molecules.*

Color: white
Luster: dull, earthy
Habit: massive, fibrous with tubular morphology
Hardness / Specific Gravity: 1.0 - 2.0   / 2.0 - 2.2

      Optics: (+)
          Pleochroism: none
          Refractive indices: α= 1.539, β= 1.589, γ= 1.569
          Birefringence: moderate
          Extinction Angle: 0, 90°

      Crystallography: Monoclinic
          c= 5.242

Distinguishing features:
      The high presents of Al. This Aluminum Silicate may appear in fibrous form with a tubular
      morphology.

Occurrence:
      See kaolinite

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KAOLINITE

Class: Phyllosilicate
Group: Clay Mineral

Kaolinite Al2 Si2 O5 (OH)4

Color: white; may be colored by impurities.
Luster: dull, earthy
Habit: Usually white earthy masses may be colored by surrounding material. Crystals are usually hexagonal plates.
Hardness / Specific Gravity: 2 - 2.5 / 2.6 - 2.7
Cleavage / Fracture: {001} perfect, rhombic or hexagonal plates

Optics: (+)
pleochroism: none
Refractive Indices: α=1.539, β=1.589, γ=1.589
Birefringence: moderate (0.045)
Extinction Angle: 0, 90^0

Crystallography: Triclinic; -1
P-1; a=5.27; b=9.12; c=18.85; β=100^{0}0'   a:b:c=0.578:1:2.067
Z= 4. d's: 9.34(10), 4.66(9), 3.12(10), 2.48(7), 1.87(4)
Orientation: 2V=6-30^0, Z=b, X$\perp${001}; r>v

Composition:

MgO 31.7, SiO2 63.5, H2O 4.8
Al or Ti may substitute for Si. Fe may substitute for Mg. Cleavage: {001} perfect. Thin flexible folia. Care should be taken during TEM analysis. The chemical analysis may yield a breakdown similar to chrysotile asbestos and the platy aggregates can easily lie upon one another in a way as to appear tubular in morphology. The distinguishing feature is the hexagonal diffraction pattern quite unlike that of chrysotile. Usually found in clay like masses.

Distinguishing features:

infusible, insoluble. When moistened with cobalt nitrate and ignited it takes on a bluish color.

Occurrence:

The chief constituent of kaolin or clay. It is always a secondary mineral formed by the hydrothermal alteration of aluminum silicates, particularly feldspar.

PALYGORSKITE

CONFIDEN : :

Class: Phyllosilicate
Group: Clay Mineral

Palygorskite $(Mg,Al)_5 (Si,Al)_8 O_{20} (OH)_2 \cdot 8H_2O$

Color: white, gray
Luster: dull, earthy
Habit: massive, fibrous with tubular morphology
Hardness / Specific Gravity: 1.0 - 2.0 / 2.0 - 2.2

Optics: (+)
Pleochroism: none
Refractive indices: α= 1.539, β= 1.589, γ= 1.589
Birefringence: moderate
Extinction Angle: 0, 90°

Crystallography: Orthorhombic
Pn: a= 12.725; b= 17.872; c= 5.242 β= 92.23°

Distinguishing features:
The high presents of Al. This Magnesium Aluminum Silicate may appear in fibrous form
with a tubular morphology.

Occurrence:
See kaolinite

TALC

Class: Phyllosilicate
Group: Clay Mineral

Talc Mg3 Si4 O10 (OH)2

Color: white, gray or pale green; often stained reddish and translucent.
Luster: dull, pearly on cleavage surfaces.
Transparency: translucent
Habit: Usually granular or foliated masses; crystals and fibers are rare.
Hardness / Specific Gravity: 1 (will make a mark on cloth) / 2.7 - 2.8
Cleavage / Fracture: basal, perfect {001}

Optics: (+)
Pleochroism: none
Refractive Indices: α=1.539, β=1.589, γ=1.589
Birefringence: moderate (0.045)
Extinction Angle: 0, 90°

Crystallography: Monoclinic; 2/m
C2/c; a=5.27; b=9.12; c=18.85; β=100°0' a:b:c=0.578:1:2.067
Z= 4. d's: 9.34(10), 4.66(9), 3.12(10), 2.48(7), 1.87(4)
Orientation: 2V=6-30°, Z=b, X$\perp${001}; r>v
Composition: MgO 31.7, SiO2 63.5, H2O 4.8
Al or Ti may substitute for Si. Fe may substitute for Mg.
Cleavage: {001} perfect. Thin flexible folia.

Distinguishing features:
Greenish white color, extremely soft, soapy feel. Flexible but not elastic.

Optically:

Usually tabular with hexagonal or rhombic plate outline. Foliated. Sometimes massive, talc may appear as fine thin ribbons under PLM. TEM usually shows platy or rhombic outline with near perfect cleavage. Diffraction analysis yields distinct hexagonal patterns, which remain constant during tilting.

Care should be taken during TEM analysis. The chemical analysis may yield a breakdown similar to chrysotile asbestos and the platy aggregates can easily lie upon one another in a way as to appear tubular. The distinguishing feature is the hexagonal diffraction pattern quite unlike that of chrysotile.

Occurrence:

Talc is formed by the alteration of magnesium silicates such as pyroxenes, amphiboles and olivine. Usually found in metamorphic rock. Known as *soapstone* in its massive form.

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WOLLASTONITE

Class: Inosilicate
Group: Pyroxenoid

Wollastonite Ca Si O3

Color: colorless or white to gray
Luster: vitreous; pearly on cleavage surfaces; silky when fibrous
Transparency: subtransparent to translucent
Habit: Triclinic; (-1)
Hardness / Specific Gravity: 4.5-5.0 / 2.8-3.0
Cleavage / Fracture: {100} & {001} perfect, {-101} good giving splintery fragments.

Optics: (-)
$\alpha=1.620$, $\beta=1.632$, $\gamma=1.634$
$2V=40°$, Y near b, $X^{\wedge}c=32°$

Crystallography:
P-1; a=7.94; b=7.32; c=7.07; $\alpha=90°2'$, $\beta=95°22'$, $\gamma=103°26'$; a:b:c=1.084:1:0.966
Z=6. d's: 3.83(8), 3.52(8), 3.31(8), 2.97(10), 2.47(6)

Composition:
CaO 48.3%, SiO_2 51.7% for pure $CaSiO_3$. Some Fe, Mn, & Mg may replace Ca. Pseudowollastonite is stable above 1120°C, has a larger unit cell (Z=24) as compared to (Z=6) of wollastonite.

Distinguishing Features:
Fusible at 4 to a white almost glassy globule. Soluble in HCl. Distinguishable by two perfect cleavages of about 84°.

Sample Reports and Worksheets

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Standard Operating Procedure

EMSL Analytical, Inc.

Quality Control Program

Standard Operating Procedures
Quality Control Program
Asbestos Laboratory

Original Date: August , 2000
Revision Date: February, 2001

EMSL Analytical Inc., Quality Assurance Department

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Table of Contents

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1.0   INTRODUCTION

The process of Quality Control analysis provides a tool for measurement of analytical performance.

EMSL utilizes a corporate controlled program which manages, tracks and stores the quality control data produced by the asbestos laboratory. The laboratory enters QC data into Excel worksheets which then provides information including:
   ♦ Acceptance/rejection of replicate and duplicate data
   ♦ Measures performance trends
   ♦ Measures accuracy of method and analysts
   ♦ Institutes upper and lower control limits
   ♦ Provides acceptance/rejection criteria for calibration measurements
   ♦ Manages and maintains records of QC and calibration measurements
   ♦ Records contamination control events
   ♦ Monitors Corrective actions
   ♦ Generates Control Charts
   ♦ Tracks QC frequency and rates quantity

EMSL performs quality control measurements as per the procedures documented in the Quality Assurance Program in EMSL.QAASB101.

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2.0 PROCEDURES

2.0 Procedures

2.1 Polarized Light Microscopy

IMPORTANT NOTE: EMSL QC procedures call for the use of the PLM
Accuracy program to track analyst accuracy. The CV spreadsheet is no
longer utilized.
The standard operating procedures for the CV spreadsheet is included in
this section as an option to the laboratories that wish to use this program
in addition to the PLM Accuracy.

PLM Accuracy

To determine the accuracy of the PLM analyst, data is generated using
reference standards with known concentrations. These are (generally) samples
from past Proficiency Testing (PT) Rounds such as those distributed by NVLAP
and the AIHA or other state programs.

Compile a set of standards for each of four ranges: 1-3%, 4-10%, 11 - 50%, and
50 - 100% asbestos. Build up your library of test samples over time so that there
are at least 5 or 6 samples in each range.

Analyze at least one standard daily. Pick the sample at random unless you need
to concentrate on a particular range.

The Program
The 'known' result is placed in the column – Reference Values – from reports of
analysis from the PT provider.

Assessment of Quantitative Performance
The program calculates the % recovery of the analysis results using:

$$\%Recovery = \frac{analysis\ result}{reference\ value} \times 100$$

The spreadsheet determines in/out of control using the acceptance criteria:

Concentration Ranges	Acceptance Criteria (%)
1-3	0→400
4-10	0→300
11-50	30→170
51-100	40→160

Of course, the "best" recovery is 100%.

An average % recovery is displayed at the bottom of the table.

<u>Assessment of Qualitative performance</u>

Qualitative IN/OUT of Control is determined by the person entering the data. If you got the asbestos type correct then IN otherwise OUT.
Also "Asbestos" vs. "No Asbestos Detected" is OUT of control as well.

Using the Program
Each analyst should maintain his/her own "PLM Accuracy.xls" spreadsheet.

PLM Accuracy is used as the template then saved in the following format: "PLM Accuracy PK.xls" with PK being the analyst's initials.

Data entry should occur only in the yellow shaded areas (with the exception of the one time name change customization)

NOTE: This spreadsheet does not get started new each month. It should be a revolving average of the most recent 20 analyses in each range.

Once you have 20 values for each range, you should always replace the oldest piece of data with the new.

Each month the analyst should print each of the 4 pages of the spreadsheet.

The print range is already selected to include only up to and including the Quantitative IN/OUT of control column.

If your computer prints out more or less of these columns, adjust some of the column widths.

NOTE: Do not print out the chart as a separate page. It is meant to be viewed with the corresponding data.

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PLMCV.xls (Optional)

4 samples are analyzed, each 20 times, in concentration ranges from<5%, 5-10%, 10-20%, >20% (or similar combinations). Care is taken to minimize the bias which may occur when an analysts becomes familiar with any given sample. *Sample numbers should be periodically changed or samples rotated.*

Permanently mounted slides make this exercise convenient by providing sample consistency (ie: as aliquots are removed for analysis from the original sample, asbestos concentrations may change), are easier to use by the analyst and help control use of the limited availability of PT samples.

Concentration ranges used are chosen by the laboratory manager and depend on availability of known samples. In all cases, ranges must accurately represent the varied concentrations encountered by the analysts in true samples. This sample pool is to include the lower-more difficult ranges (1-10%) of concentration.

Precision

Using 20 data points, a coefficient of variation is generated for concentration ranges selected and is calculated as:

CV= Standard Deviation/Mean

This data is stored in the analyst files as part of documented performance, qualifications and practical training. The Laboratory Manager and QA Manager periodically review the data for information on performance and to monitor any trends.

Accuracy

In addition to the CV, Percent Recovery is also calculated for each pair of data. This provides a measure of analysts accuracy.

This information is calculated:

% Recovery= (sample result / known value) X 100

This CV and % Recovery study is intended to characterize the ability of the analyst precision and accuracy when determining the concentration of asbestos, not type. Performance of asbestos identification is monitored with the daily

analysis of standard reference slides (see section PLM2.0 Calibration, EMSL.QAASB 101.0) and proficiency testing samples.

Frequency
This is normally a one time study, although may be extended to improve performance when needed.

The Program
Spread sheet PLMCV.xls, contain tabs (worksheets) labeled CV1 → CV8. Each of these tabs represent a different sample. There are 6 tables in each, for 6 different analysts. Graphs below each table show % recovery data for each analysts. The data is plotted about 100, which is the 'perfect score' (100% recovery).

The last tab summarizes the CV and % recovery data for each sample- for each analyst as well as overall laboratory (average of analysts).

Acceptance Criteria
% Recovery:
The limitations of the method accuracy are well known. The % recoveries are expected to be relatively high, especially in the lower concentrations. The general acceptance limits for samples of various concentrations are listed here:

Table 1.0 method acceptance ranges

% area asbestos	% recovery
1	50-300
5	20-180
10	50-150
20	50-150
30	65-135
40	75-125
50	80-120
60	85-120
70	85-115
80	85-115
90	85-115
100	85-115

These general acceptance limits are found in the method – EPA/600/R-93/116 and are to be used as guidelines only. Laboratories should adjust the acceptance criteria using historical performance data, practical and logical analysis and prudent evaluation.

CV:
True acceptance criteria for the measurement of CV has not yet been determined as of the date of this document. Generally, CV's should not exceed .40.

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PLMQC.xls

Quality Control in the PLM laboratory consists of interanalyst, intraanalyst and interlaboratory analysis. (seo EMSLQAASB101.0). The program tracks and graphs the data using a number of worksheets for data entry and statistical comparison, as described below. The program does not track the results from daily standard analysis, blank analysis or microscope calibrations.

Data Input- this simply tracks the data as generated. This includes inter and intra analysts and interlaboratory. Information is hard entered in this spread sheet – (no calculations are performed).

Report-the data entered into 'Data Input' is compared to each other using the "R-value" analysis. Here, the program takes the difference of the 2 pieces of data and divides it by the average, as explained here:

For intra analysts $\qquad R = |(A-B)/((A+B)/2)|$

For inter analysts $\qquad R = (A-B)/((A+B)/2)$

Where: $\qquad R$ = the measure of variance for the analysis

$\qquad A$ = the value of the first analysis in %

$\qquad B$ = the value of the second analysis in %

Notice we use absolute values when comparing intra analyst data. We are concerned only with the true difference of the data, rather than if they are on one side or the other. (bias up or down). The pure difference of the data characterizes the analysts analyzing their own sample and the type of bias is not of interest.

Charts(inter/intra)- the R-value of each data pair is graphed about zero (a 'perfect score'). The upper control limit is placed at 1 (see acceptance criteria below).

Although these graphs have been set up to allow for the plotting of variance data below 1, this will not occur for the intra analysts report because we use absolute values for the analysis of intra analyst data.(see discussion above),

Same Analyst/Different Analyst/Analysts 1, etc. -These spreadsheets simply organize the data into the applicable QC type and make for easy graphing.

Acceptance Criteria

The criteria used in the program are as follows:

$\qquad$ For intra analysts, $R > 1$ – FAIL or when asbestos identification errors occur

For inter analysts, R>1 or R< -1.0

This pass/fail criteria is used for the analysis of paired data as they relate mathematically to the QC program. It is important to keep in mind, criteria for the correct identification of asbestos is established in the QA manual – EMSLQAASB101.0 and the PLM SOP. This data is not collected by this QC program and must be tracked 'by hand'.

Oilcal.xls

This spread sheet tracks refractive index oil calibration. Using dispersion colors and their matching wavelength, the refractive index of the oil is measured.

The user enters the following data:
- Manufacturers calibrated refractive index
- Oil Identification (RI, lot number, etc.)
- Dispersion Color observed
- Matching refractive index. This information is found in the reference "Refractive Index Liquid Calibration Using Optical Glass Standards" Shu Chun Su, PhD.
- Change in refractive index per degree Celsius. This information is supplied by the manufacturer, found on the bottle label
- Temperature at the time of calibration
- Refractive index measured of the oil

The program calculates the true, temperature corrected refractive index.

Acceptance Criteria
The difference between the calibrated refractive index and the labeled refractive index must be < 0.004.

2.2 TEM   CONFIDENTIAL

TEMCAL.xls

EDXA Resolution Check

Resolution is very important because may of the peaks of interest for asbestos samples are very close in energies. If, for example your resolution is poor, a broad magnesium peak could hide a sodium peak that might be present right next to it leading you to misidentify crocidolite fibers

The resolution of the Xray detector is measured using a grid with some form of Manganese deposited on it.

The resolution of a particular elemental peak is defined as the width of that peak at half of its maximum value (full width half max or FWHM). What we like to see is *very tall and very narrow peaks.* The wider the peak the poorer the resolution.

To perform the calibration, collect a spectrum from the Mn calibration grid for 200 to 300 seconds. You want a lot of counts in the peak to aid in the statistics.

Once you have finished collecting, move the cursor to the left of the peak and on a channel that is representative of the background, record the counts in that channel into the TEMCAL spreadsheet (resolution page). Do the same for the background level to the right of the peak.

Next you are ready to record the channel by channel counts in the Mn peak. Start at the left side of the peak and move the cursor channel by channel left to right, entering the count information into the spreadsheet as you go. Each channel should be set to 10 KeV.

The exact start channel is not critical as the very beginning and end data are not used in the calculations. Likewise it is not necessary to enter values for all 40 spaces on the spreadsheet.

Frequency
Resolution is measured semi-annually

The Program
The program determines the channel with the *max value (after it subtracts the* background values). It then determines at what channels the half max values fall on. In actuality these values fall in between two channels. The program is smart enough to perform the extrapolation and determine the exact resolution.

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Acceptance Criteria

The resolution must be <175eV and the value for the sum of the resolution and the variation (2 σ) that is < 180eV.

The spreadsheet will perform all the calculations necessary and tell you if you PASS or FAIL. The results also are automatically placed in the report page of TEMCAL.xls. If the results show a FAIL then recollect the spectrum and try again making sure to minimize background noise by:

- Collecting spectrum from the center of the grid
- Make sure the tilt angle is correct
- Select a collection area from center of grid opening
- Collect long enough to collect 10,000 or more total counts or more in the Mn peak

If after multiple tries the resolution is still greater than 175 ev then the detector may need to be recycled or replaced. Contact Bruce Falseit or your Regional Manager.

NOTE: The capabilities of the various lab's EDS systems varies. This program provides a lowest common denominator approach to obtaining a resolution value. If your system is capable of performing some or all of these calculations directly then many of the above steps can be reduced or eliminated however please get prior approval from the QA department.

K Factor Determination using SRM 2063

In order to perform any kind of Quantitative X-Ray analysis we need to know the sensitivity of the detector to the elements of interest. These sensitivities are expressed relative to some other element (typically Silicon) hence the term relative sensitivity factors or K Factors.
The Xray detectors we use are Silicon/Lithium (SiLi) detectors with Beryllium windows. These detectors are only sensitive enough to detect elements down to and including Sodium. They are much more sensitive to heavier elements such as Iron. This is why, given a spectrum with equal Mg and Iron peaks, we cannot assume that there is 50% Mg and 50% Fe in the sample. In fact there is probably much more Mg since we know that many more of the Mg Xrays are being missed by the detector than from Fe.

To perform this calibration we need a standard with known quantities of various elements. SRM 2063 is a thin glass film produced by NIST, with known quantities of Mg, Si, Ca and Fe.

Collect for 200-300 seconds in order to get good counting statistics. (10,000 counts in the Silicon peak is the typical rule of thumb).

Enter the background subtracted counts for Mg, Si, Ca, and Fe into the TEMCAL spreadsheet (Kfactor page).

Print, date and sign spectrum.

Repeat for a total of twenty repetitions.

Frequency
These measurements are performed semi-annually

The Program
On the K Factor page of TEMCAL enter the background subtracted counts for Mg, Si, Ca, and Fe into the TEMCAL spreadsheet (K-factor page).
The program looks at the total counts in each peak compared to the known percentage of each element and calculates a K Factor for each element (relative to Silicon) for each run.

Once you have entered in all data for all six runs the program calculates an average K Factor value for each element relative to Silicon and also the K Factor of Mg to Fe. This K Factor is calculated as:

<u>K factor for Mg, Ca and Fe to Si</u> = % of element (Mg, Ca or Fe) / % of Silica x counts of Silica / counts of element being measured.

<u>K factor for Mg to Fe</u> = % Mg / % Fe x counts of Fe/counts of Mg

Certified Concentration % by weights:

Mg	7.97
Si	25.34
Ca	11.82
Fe	11.06

Acceptance Criteria
K Factor of Mg relative to Fe must be 1.5 or less
K Factors of Mg relative to Si must be between 1.0 and 2.0
K Factors of Ca relative to Si must be between 1.0 and 1.75
-and-
K Factors to a precision (2σ) within 10% relative to the mean value obtained for Mg, Si, Fe

Mapping of abnormal xray
XXXX

K Factor Determination using Albite

To perform the calibration for the elements Al and Na, we need a standard with known quantities of these various elements. SRM 2063 is great for determining the relative sensitivities for most elements that we encounter during asbestos analyses but not for aluminum and sodium. This is why we perform the K Factor determination with an albite standard that has been characterized by microprobe analysis.

With an albite standard, collect a spectrum for 200-300 seconds in order to get good counting statistics. (10,000 counts in the Silicon peak is the typical rule of thumb).

Enter the background subtracted counts for Na, Al, and Si into the TEMCAL spreadsheet (Kfactor page).

Print, date and sign spectrum.

Repeat for a total of twenty repetitions.

Frequency
These measurements are performed semi-annually

The Program
On the K Factor page of TEMCAL enter the background subtracted counts for Na, Al, and Si peaks into the TEMCAL spreadsheet for albite (K-factor page). The program looks at the total counts in each peak compared to the known percentage of each element and calculates a K Factor for each element (relative to Silicon) for each run.

Once you have entered in all data for all twenty runs the program calculates an average K Factor value for each element relative to Silicon

K factor for Al to Si = % of element (Al) / % of Si x counts of Si / counts of Al.

K factor for Na to Si = % of element (Na) / % Si x counts of Si/counts of Na.

Concentration , elemental % by weights for the albite standard:
Na   8.75
Al   10.27
Si   32.07
K   0.10
Ca   0.07
O   48.76

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Acceptance Criteria
K Factor of Na relative to Si must be between 1.0 and 4.0
K Factor of Al relative to Si must be 1.0 and 1.75
-and-
K Factors to a precision (2σ) within 10% relative to the mean value obtained for Al, and 20% for Na

Magnesium and Silicon Detection

This is another test of the Xray detector's performance. If a detector is performing poorly it will not be able to detect Xrays from very small fibers above background levels.

Collect a spectrum from a single fibril (< 0.05 micron width) of NIST traceable Chrysotile.

Magnesium and Silicon peak should be resolvable.

Frequency
Mg, Si are measured semi-annually

The program
Enter the information into the TEMCAL spreadsheet (Na Mg Si page). This automatically reports the information on the "Report" page.
Print, sign, and date the spectrum and keep with monthly QC report

Acceptance Criteria
Resolvable Magnesium and Silicon peaks

Sodium Peak Sensitivity

The Xray detectors we use are Silicon/Lithium (SiLi) detectors with Beryllium windows. These detectors are only sensitive enough to detect elements down to and including Sodium. Since sodium is an element of interest in asbestos analysis it is important for us to periodically determine whether or not our detectors are actually detecting it.

It is not enough that we can see the sodium peak. It must be there with enough counts to be statistically significant above the background levels.

To perform this calibration we collect a spectrum from a NIST traceable Crocidolite sample. We collect for 200-300 seconds in order to get good counting statistics. (10,000 counts in the Silicon peak is the typical rule of thumb).

Frequency:
Measured Quarterly

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The Program
Once the spectrum has been collected we record the integral (whole peak) count values of the sodium peak before and after background subtraction. These values are entered in the TEMCAL spreadsheet (Na Mg Si page). The spreadsheet will perform the calculations and PASS/FAIL determination.

SODIUM PEAK IS SIGNIFICANT IF N > n + 3 (SQUARE ROOT OF n x 2)

   where: N = background subtracted
        counts
        n = backround
        counts

Acceptance Criteria
Sodium peak must be statistically present

    Corrective Actions
       If, on multiple attempts the results are failing make sure:

          o   collect spectrum from center of grid
          o   collect spectrum from the center of grid opening
          o   ensure proper tilt angle of specimen
          o   you are collecting a spectrum from a suitable fiber with no extraneous material around or covering it

       Once an acceptable spectrum has been produced print it out, sign it, date it, and file it with your monthly QC Reports.

       If none of the above helps your detector may need to be recycled or replaced. Call Bruce Falseit or your Regional Manager.

Magnification Calibration on Film
In order to correctly determine the size of fibers for classification it is essential to know the exact magnification of the microscope.
The procedure to determine this is fairly straightforward with the help of a magnification calibration grid. These grids are carbon replicas of a lattice of known spacing (most commonly 2160 lines per mm). Sometimes the replica is a series of lines and sometimes a series of squares (waffle pattern). Both are identical for our purposes.
Take a picture of the standard at both 10,000 and 20,000 times magnification.

Eucentricity and critical focus are important!
When the film has been developed measurements are taken off the negative and recorded into the TEMCAL spreadsheet.

Frequency: Monthly

The Program
On the magnification page of the TEMCAL spreadsheet enter the date, negative number and measurement information. Measure the distance across numerous squares or lines for increased accuracy. Enter the TOTAL distance and the TOTAL number of lines being measured. The program calculates the size of each square or line and then the exact magnification.

Acceptance Criteria:
Magnifications are charted over time. The variation of calibration data points is managed by the spreadsheet. The criteria is defined as 2 X the standard deviation of the past measurements to date. This number must not be >5% of the mean.

Magnification Calibration on Screen
Most of out laboratories are using JEOL 100CXII microscopes with phosphorescent screens that are not very condusive to on screen measurements. The small and large circle are the 0.5 micron and 5 micron measuring aids respectively. By measuring how many layer lines of the mag cal grid fit in these circles we can calculate the on screen magnification. This magnification is always less than the magnification at the film level.

In order for this calibration to be accurate it is necessary to know the exact ACTUAL diameter in mm of both the small and large circle. The actual size of the small circle is especially important as this is the 0.5 micron (minimum size to be counted) visual aid during analysis.

If these values are not known it will be necessary to remove the glass viewing screen and take these readings. The diameters are entered into the magcal section of the TEMCAL spreadsheet. This need be done only once. (unless the screen is replaced).

Frequency: Monthly

The Program
The on screen measurement data can be recorded in the photo log with the corresponding "on photo" information. Just a note saying for example "10.5 lines in large circle at 20,000X"
This information can then be entered into the spreadsheet at the same time you are entering the "on film" measurements. In the "On Screen" portion of the

Magnification page of the TEMCAL spreadsheet, enter the Date and measurement info (number of lines seen in the large circle). The program will then calculate the actual screen magnification and percent difference from the average and from the target.

Acceptance Criteria:

Magnifications will be charted over time. The variation of calibration data points is managed by the spreadsheet. The criteria is defined as 2 X the standard deviation of the past measurements to date. This number must not be >5% of the mean.

The charts

Utilizes three types of graphs for each magnification (10,000x both on screen and film, and 20,000x both on screen and film)

- Plots the 'Actual' magnification obtained on the given date against the 'Target Value' magnification for the specified magnification.

- Plots percentage of variance:
  -% away the 'Actual' value for that day is, against the 'Target' value.
  -% away the 'Calculated Mean' is from the 'Target' value.

  NOTE: These two lines should show a similar trend/pattern when plotted, as they directly correlate with each other

- These Control Charts use the acceptance/rejection criteria:
  (2 Standard Deviations must be < 5% Mean)
  Here 5 % of the cumulative mean to date is plotted as one line and 2SD is plotted as another. The 2SD line should always be "below" the charted line showing the value is "less than" that of 5 % of the mean

Spot Size Measurement

Because it is sometimes difficult to isolate a fiber for Xray analysis, the TEM scope must be able to achieve a very small spot. Some of the older scopes do not have a double gap pole piece in the condenser lens and are therefore unable to meet the spot size requirements. In order to measure the spot size we take a photo of spot size 3. After developing we measure the diameter of the spot directly from the negative.

Frequency:
An electron micrograph verifying the calibration must be recorded yearly.

The Program:
On the "magnification page of the TEMCAL spreadsheet go to the spot size section.
Enter the date, the most recent calculated Magnification and the mm diameter measurement taken from the negative
The program will then calculate the exact spot size nanometers.

The Charts
These Control Charts use the acceptance/rejection criteria:
2 Standard Deviations MUST BE < 5% Mean
Here 5 % of the cumulative mean to date is plotted as one line and 2SD is plotted as another. The 2SD line should always be "below" the charted line showing the value is "less than" that of 5 % of the mean.

Acceptance Criteria: In order to meet AHERA requirements the spot size used for X-Ray Analysis must be 250 nm of less This is because it is sometimes difficult to isolate the fiber of interest. The variation of these measurements is tracked over time and managed by the spreadsheet. The acceptance is defined as 2 X the standard deviation of the past measurements to date. This number must not be >25% of the mean.

Camera Constant Determination
Similar to magnification calibration, the camera constant is basically a magnification determination when the scope is in diffraction mode. By taking a diffraction pattern of a sample with a known d-spacing (gold) and measuring the spacings on the negative we can accurately determine the magnification or camera constant.

Frequency
Monthly

The Program

Once you have developed the negative you can measure the diameter of the various rings in mm.

On the camera constant page of the TEMCAL spreadsheet enter:
-The date the picture was taken
-The negative number
-The camera length the pictures were taken at
-The mm measurements of the diameter of the first ring (in three directions) and of the 3rd ring (also in three directions).

Multable measeruement sare made to takc in to accoutn any stigmatism
that may be present.3 on all 3 rings? or one on all 3 rings.
The program will then calculate the camera constant

Charts

Utilizes two types of graphs:
Here graphs reflect the line measurement and if applicable the on-screen measurement acquired on the given dates.

    I)   Plots percentage of variance
        -% away the 'Actual' value for that day is, against the 'Target' value.

    II)   These Control Charts reflect acceptance/rejection criteria.
        (2 Standard Deviations MUST BE < 5% Mean)
        Here 5 % of the cumulative mean to date is plotted as one line and 2SD is plotted as another. The 2SD line should always be "below" the charted line showing the value is "less than" that of 5 % of the mean.

Acceptance Criteria
The variation of the measurements is tracked over time and managed by the spreadsheet. The acceptance is defined as 2 X the standard deviation of the past measurements to date. This number must not be >5% of the mean.

The electron microscope should be able to obtain diffraction patterns *from single* fibrils of chrysotile without excessive beam damage. From a standard made of NIST traceable Chrysotile (such as 1866), obtain diffraction patterns from ten individual single fibrils of chrysotile and observe the diffraction patterns for 15 seconds.

Beam Dose

Frequency
Semi-Annually

The Program

On the beam dose page of the TEMCAL spreadsheet enter:
-The date
-NIST std used
-Number of patterns obtained
-Number of patterns remaining after 15 seconds
-Negative number of SAED pattern
-Negative number of image

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Acceptance Criteria
Chrysotile fibril must remain visible for 15 seconds.

Plasma Asher
The ashing step serves two main purposes. One is to remove any organic material from the surface of the filter to make the asbestos analysis easier. The *check what the actual program* second purpose is to actually ash the top 5-10% of the MCE filter (also organic) away to reveal or expose any asbestos fibers that may have been covered over by the clearing (collapsing) process. Too little ashing and some or all of the fibers don't get exposed. Too much ashing and you run the risk of fiber loss and also the surface of the filter becomes so pitted that the prep suffers from excess carbon film breakage or a filter surface that is too rough and grainy to analyze.

To perform this calibration ash pre-weighed MCE filters for a set period of time. After ashing we weigh again to see what percentage has ashed away.

Frequency
These measurements are performed quarterly

The Program
On the Plasma Asher page of the TEMCAL spreadsheet enter:
-Date
-Ash Time
-Gravimetric Data

The program calculates the weight loss and also the time then necessary for a ten percent ash.

Acceptance Criteria
Ashing times are measured to determine the time for a 10% ash.

TEM QC
1876b.xls

A measure of analysts accuracy is performed with the analysis of the NIST traceable Standard Reference Material (SRM) 1876b. Analysis is done on these samples following the NIST counting rules submitted with the SRM. – "National Institute of Standards and Technology Certificate Standard Reference Material 1876b. A Chrysotile Asbestos Standard Reference Material for Transmission Electron Microscopy". The analyst counts 12 different grids and enters the data into the spread sheet..

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Frequency:
All active analyst (those qualified to perform analysis-see EMSL.QAASB module B 1.0) must analyze 1876b at least once a year.

The Program
The program monitors and tracks the results for each analyst. It calculates the mean, standard deviation and coefficient of variation. A trimmed mean value is obtained from the 12 results by averaging 10 counts after the highest and lowest counts are eliminated from the 12 counts. This trimmed mean value is then compared to the lower limit and upper limit values, as described below.

Acceptance Criteria
From the data found in the SRM certificate, the actual value of the sample is 18.2 structures per 0.01 mm^2 ± 3.5 structures. Guided by the recommendations of the NVLAP program, we use 80% of the lower limit (at 18.2 − 3.5 structures) and 110% of the upper limit (at 18.2 + 3.5 structures) as our acceptance criteria.

Therefore:

<u>Target Value</u> -   18.2 structures per 0.001mm^2 (s / 0.001 mm^2) or **1820 structures per mm^2**

<u>Lower Limit</u> -   18.2 − 3.5 = 14.7 (actual lower limit)
14.7 x 80% = 11.76 structures per 0.01 mm^2 or **1176 per mm^2.**

<u>Upper Limit</u> -   18.2 + 3.5 structures = 21.7 (actual upper limit)
21.7 x 110% = 23.87 structures per 0.01 mm^2 or **2387 per mm^2.**

<u>TEMQC.xls</u>
Some of the most useful information about the performance of an analysts is found with sample reanalysis. This is done using a selection of types of reanalysis. These include:

    Intra analyst – same analyst
    Inter analyst - different analyst
    Inter analyst verified – different analyst using verified counting technique
    Inter laboratory (Round Robin) – different laboratory

Details on these various types of QC analysis can be found in EMSL.QAASB 101.0 Module B, 6.2.1 and EMSLTEMSOP.200.0

Frequency:

The frequency of QC analysis is based on requirements by accrediting authorities and GLP (Good Laboratory Practice). These are:

    Intra analyst – 2% of sample volume
    Inter analyst – 4% of sample volume
    Inter analyst verified – 1% of the grid openings analyzed
    Round Robin - .5% of sample volume

The Program
Data Input – All results of reanalysis is entered in this spread sheet

Inter/ Intra analyst , Interlaboratory

Reanalysis data is evaluated by comparing the differences of the 2 results and dividing by the average – called Variance (or R value). It compares the data for each analyst being checked, with a second analyst.
Calculations are

For intra analysts $R = |(A-B)/((A+B)/2)|$

For inter analysts $R = (A-B)/((A+B)/2)$

Where: A = the result of the analyst being checked

             B = the result from the other analyst for the same grid opening

Notice we use absolute values when comparing intra analyst data. We are concerned only with the true difference of the data, rather than if they are on one side or the other. (bias up or down). The pure difference of the data characterizes the analysts analyzing their own sample and monitoring the type of bias is not necessarily of interest.

Chart: The data is charted for each analyst, for each QC type. Sample number is the x-axis. Variance is charted on the y –axis. This graph does not demonstrate pass of fail, it rather provides a statistical tool for evaluating laboratory precision.

Verified Analysis
The evaluation of the data for verified results involves the comparison of the data generated by the analyst and a QC analyst. Any disparity is resolved with a confirmatory analysis. The confirmed result is used generate information of the analysts performance by tracking the %'s of True Positives (TP), False Negatives (FN) and False Positives (FP). The spread sheet calculates and graphs the information.

Example:

Analyst 1 – counts 10
Analyst 2 – counts 15
Confirmed results – counts 12

Evaluation: Analyst 1 =   83% TP (has counted all 10 of the 12 fibers in the
                            sample –but missed 2)
                            17% FN (has missed 2 of the 12 fibers in the sample)
                            0% FP (has not counted any more than what is
                            actually there)
                            Should be_(83% TP
                            17% FN)
            Analyst 2 =   80% TP (over counted by 3 - not all of the 15 counts
                            are true- 25% is false, therefore the total count is only
                            '75% true')
                            0% FN (has not missed any fibers)
                            20% FP (over counted 3)

Asbestos Identification errors are also entered into the spreadsheet for tracking.
A % of the misidentified count(s) is assigned and displayed. This data is hard
entered and is tracked by 'hand only'

Acceptance Criteria

Verified Analysis:
The acceptance criteria used for analysts performance are:
>= 80% True Positive
<= 20% False Negatives
<= 10% False Positive

Important note: the program does not flag missed Identification as a 'failure'..
This must be noted by the laboratory Manager (see EMSL.QAASB101.00)

Inter/ Intra analysts, Intralaboratory:
The program calculates the pass/fail of the 2 pairs of data using:
If the mean of the recount (average) is < 5 structures, acceptable
agreement between the 2 counts is = +/- 1 structures

If the mean of the recount (average) is 5-20 structures, acceptable
agreement between the 2 counts is = +/- 2 structures

If the variance is > 20 structures, the acceptable agreement between the 2
counts is = +/- 3 structures

The program performs the above calculation and assigns a pass or fail the worksheet column. This information is not displayed graphically.

2.3 Phase Contrast Microscopy

PCMCV.xls

Accuracy
A coefficient of variation is generated for use in the calculation for the acceptance criteria.

Daily reference sample data for each analyst is collected and tracked in the EMSL Monthly QC report. Analysis is performed using past proficiency testing (PAT) rounds. For each set of 20 data points, a coefficient of variation (CV) is determined for each individual analyst in fiber ranges of:

> 5-20 fibers/100fields
> 21-50 fibers/100 fields
> 51-100 fibers/100 fields

As these standard samples are analyzed, current data replaces the oldest data point.

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Precision
PCMQC.xls
Sample reanalysis is performed as intra analyst (same analysts) QC at the rate of 10% of sample volume. The spreadsheet compares the 2 pieces of data and calculates the pass/fail criteria following NIOSH 7400 requirements as follows:

Pass - If the absolute value of the difference between original and QC analysis (in f/mm2) is less than or equal to the value calculated by the constant (2.77) multiplied by the average of the square root of the original and QC fiber counts times the calculated CV value divided by 2.

$$\left| \sqrt{original f \ / \ mm^2} - \sqrt{qcf \ / \ mm^2} \right| \leq 2.77 \left(\frac{\sqrt{f \ / \ mm^2} + \sqrt{f \ / \ mm^2}}{2} \right) \frac{CV}{2}$$

Fail - If the absolute value of the difference between original and QC analysis (in f/mm2) is greater than the value calculated by the constant (2.77) multiplied by the average of the square root of the original and QC fiber counts times the calculated CV value divided by 2.

$$\left| \sqrt{original f \ / \ mm^2} - \sqrt{qcf \ / \ mm^2} \right| 2.77 \left(\frac{\sqrt{f \ / \ mm^2} + \sqrt{f \ / \ mm^2}}{2} \right) \frac{CV}{2}$$

3.0 DATA AND REPORT ORGANIZATION

Introduction
The manner by which the final Monthly Report is organized plays an important role in the overall Quality Control plan. The presentation of the data must be arranged so as to provide the reader with a view of the laboratories overall performance.

Table of Contents:
The Table of Contents describes the order of the QC information. This page is placed after the cover page.

QC Summary Report:
This page is the key section of the report. It summarizes all of the QC activities for the month and keeps a tally on the performance of the lab (and analysts) over the year. It provides a 'snap shot' of the laboratories compliance with QC frequency and shows if the data is within established acceptance criteria.

Data is entered into the shaded areas of the spreadsheet only

There are number of issues that must be understood when using this report. These include:

1. QC Credit
The 'credit' for a QC analysis belongs to the original analyst. For example: John performs 10 reanalysis on the samples originally analyzed by Mary. Mary's total QC samples is 10, Johns is 0.

2. The Assignment of Outliers
The assignment of an outlier (same as failure in spreadsheet) must be determined by the Laboratory Manager. Which analyst has made the error is based on an evaluation of the data, an analysis by a third party, etc (see section 10.3 Corrective Action in the QA Manual for additional information).

3. Defining Outliers
The QC program uses a simple statistical measure of relative difference (R value). This is labeled Variance on the spreadsheets (see PLM and TEM QC sections above for more detail).
In the interest of simplicity, we have chosen this method of data evaluation. However, when applying this method to the PLM QC, paired results at the lower concentrations, do tend to 'fail'. Using common sense, the lab Manager should make a careful evaluation of the failure and make note on the spreadsheet if the failure is or is not justified, for the questionable cases. For example, results of 2 and 7% fail the QC check. While the difference in these 2

results should be flagged, they are not necessarily defined as a failure. See EPA/600/R-93/116 page 11, table 2-1 for guidance.

4. Measuring Qualitative Errors

For inter and intra analysis in PLM and TEM, qualitative errors must be monitored 'by hand'. The Lab Manager (or designee) reviews the report for discrepancies in asbestos type and records them on this Summary report in the # out columns.

5. Cumulative Average of the Variance

For the evaluation of QC data for PLM and TEM reanalysis, the cumulative average of the avg. variance is calculated versus the #out. This will give information on any trends that may be occurring. Keep in mind, # of outliers are (indirectly) reflected in the variance.

LAB PERFORMANCE

	Number	% Total Samples
Total Non- Analysis Errors		
All other Errors		
Total Errors		

This section tracks the total errors the laboratory may have had in the month. Average the % for each analysis (PCM, PLM and TEM) found at the end of each section.

Errors are defined as:

Non-analysis errors – these are technical type errors, such as mixing up samples (reporting results for the wrong sample), losing samples, etc. These are tracked by the lab manager or designee 'by hand' and are documented with a corrective action report.

It does not include items such as typographical errors, missed deadlines, a missing sample on the chain of custody, results faxed to the wrong client, changed turnaround times or other business related type errors. IMPORTANT: in the case where a typographical error had occurred where asbestos was detected – yet reported as ND or visa versa, a corrective action must be completed (as well as an amended report-see QA manual for more specific detail).

All other Errors - these are QC related errors. The number of 'failures' as calculated by the spreadsheets in the QC program for the interanalyst, intraanalyst, interlab and standards are tallied here. Any errors on proficiency testing (PT) results and contamination problems are also added here. Failed PT and laboratory blank results are tracked by the lab manager (or designee) by 'hand'.

% of total samples-this is the percent of total client samples, not the total number of QC samples analyzed.

PCM TOTALS

QC FREQUENCY	Samples	% of Total
TOTAL SAMPLES ANALYZED		
INTRA-ANALYST QC (1/10 or 10%)		
INTER-LABORATORY (1/200 or 0.5%)		
TOTAL QC (minimum = 10.5%)	0.00	

This measures frequency (amount) only. Notice we do not perform inter-analysts QC. (see NIOSH 7400).

 % of total – this is the % of QC performed on total client samples analyzed.

A note on Standards Analysis: the daily reference slide data is tracked in the CV section of the program and not summarized here. This data is used to generate the Coefficient of Variation for each analyst for the calculations used for acceptance/rejection criteria (see above PCM section for more detail). Daily reference slide analysis is also performed to calibrate the analyst and microscope prior to the days analysis.

LABORATORY PERFORMANCE				
INTRA-ANALYST		QC Samples	# Out	% Out

This records the data for the samples reanalyzed by the same analyst only.
Using the spreadsheet in the program ' Monthly QC for PCM Analysis', count and record the number of QC samples analyzed and number of failures.

 % out -- the % of failures on the QC samples

QC INTER-LABORATORY		QC Samples	# Out	% Out

Inter-laboratory QC are samples such as round robin analysis.
Data is entered same as above for intra-analyst.

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PLM TOTALS

	QC Samples	
		% of Total
TOTAL SAMPLES ANALYZED		
INTER-ANALYST QC (1/15 or 7%)		
INTRA-ANALYST QC (1/50 or 2%)		
STANDARDS (1/100 or 1%)		
INTER-LABORATORY (1/500 or 0.2%)		
TOTAL QC (minimum = 10.2%)	0.00	

% of Total-% of total client samples analyzed

	# Out	% Out	Cum. Avg.
BLANKS (DAILY)			
Is Monthly Data Acceptable? (Y/N)			

The lab manager (or designee) monitors the data recorded for the daily blank analysis. This data is generally recorded on a separate form kept in the PLM lab. The QC program itself does not have a spreadsheet that maintains this information.

For NYS Labs, record the Information from the blank analysis performed for NOB's (1 out of every 20 is analyzed as a blank) here also.

out - # of samples that have shown contamination

% out - % of blank samples that where contaminated (of the total blank samples analyzed)

Cum. Avg – this is the average % outliers, averaged throughout the year beginning with January. For example, April's Cum. Avg will be the average % of Jan, Feb, March and April.

LABORATORY PERFORMANCE

QC / INTER-ANALYST	Samples	Avg. Variance	# Out	% Out	Cum. Avg.

Samples – number of QC samples analyzed.
Avg. Variance – this is the average of the variance found in the 'Original' column in the spreadsheet , Monthly PLM Friable QC Summary. This information gives the overall precision of the analyst.

Note: use the true (and not absolute value) when averaging these numbers, as this will monitor any real bias an analyst may have.

 # Out – number of outliers counted from the Monthly PLM Friable QC Summary. (see also 2 and 3 in Introduction)

% Out – The % of outliers to QC sample

Cum. Avg. - this is the average of the variance, averaged throughout the year beginning with January. For example, April's Cum. Avg will be the average of the avg variance of Jan, Feb, March and April.

QC / INTRA-ANALYST	Samples	Avg. Variance	# Out	% Out	Cum. Avg.

This section is completed same as above using same analyst reanalysis

QC / STANDARDS	# STDS.	Avg % Recovery	# Out	% Out	Cum. Avg.

The data for standard analyses are taken from the EMSL PLM Accuracy spreadsheet.

 # STDS – Total number analyzed in all 3 ranges

Avg % Recovery - Average the percent recovery from all 3 ranges.

 # Out – This is the number of analysis that have 'failed' using the EMSL PLM Accuracy spreadsheet . Include both the qualitative and quantitative errors. Once again, the assignment of the outlier is determined by the lab manager (see item 2 and 3 in introduction)

% Out – The % outliers of the # STDS.

Cum. Avg - this is the average of the % recovery, averaged throughout the year beginning with January. For example, April's Cum. Avg will be the average of the % recovery of Jan, Feb, March and April.

QC / INTER-LABORATORY	Samples	Avg. Variance	# Out	% Out	Cum. Avg.

This section is completed as above using inter-lab reanalysis (and round robin data).

	Total QC Samples	# Out	% Out	Cum. Avg.
Overall PLM laboratory Performance				

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This information gives us the overall performance of the PLM lab. Add up all the data from the other sections for total QC samples and #out. Figure %out on QC samples.

Cum Avg.- this is the average of the %out, averaged throughout the year beginning with January. For example, April's Cum. Avg will be the average of the %out of Jan, Feb, March and April.

TEM AIR TOTALS

QC Frequency	Samples	
TOTAL SAMPLES ANALYZED		% of Total
INTER-ANALYST QC (1/25 or 4%)		
INTRA-ANALYST (1/50 or 2%)		
VERIFIEDS : (Inter and Intra analyses must total samples 1%)		
INTRA-LAB (1/200 GO or 0.5%)=(5/1000 Samples)		
INTER-LAB (1/200 GO or 0.5%)=(5/1000 Samples)		
BLANKS PREPPED (1/10 or 10%)		
BLANKS ANALYZED (1/25 or 4%)		
TOTAL QC (minimum = 11%)		

Note: 20% of the 1% verifieds must be performed on samples containing 6-40 structures/grid opening.

% of Total – this is the % of total client samples analyzed.

SRM 1876b (Annually)	

Record any SRM 1876b analyzed in that month here. This tracks compliance of frequency only and does not score the analysis. This is done it the next section – Laboratory Performance

LABORATORY PERFORMANCE

QC / INTER-ANALYST		Samples	Avg. Variance	# Out	% Out	Cum. Avg.

Note: Inter-analyst reanalysis is a different QC 'type' than is verified analysis. In other words, we do not consider verifieds as inter-analysts and inter-analysts as verifieds

Samples – number of QC samples analyzed.

Avg. Variance – this is the average of the variance found in the 'Original'
column in the spreadsheet , TEM QC Summary. This information gives the
overall precision of the analyst for the month.
Note: use the true (and not absolute value) when averaging these numbers, as
this will monitor any real bias an analyst may have.

 # Out – number of outliers counted from the TEM QC Summary. (see also 2 and
3 in Introduction)

% Out – The % of outliers to total QC samples

Cum. Avg. - this is the average of the variance, averaged throughout the year
beginning with January. For example, April's Cum. Avg will be the average of
the avg variance of Jan, Feb, March and April.

QC / INTRA-ANALYST	Samples	Avg. Variance	# Out	% Out	Cum. Avg.

This section is completed as above using intra-analyst reanalysis.

QC / VERIFIEDS		# Verified	% TP, FP, FN	# Out	Cum. Avg.

The information for this section comes from the spreadsheets in the verified
section of the QC program.

% TP, FP, FN – the program calculates this automatically in the Analysts
Verified Summary for Laboratory. Simply copy the data into the boxes.

 # Out – if the analyst's performance has exceeded the acceptance criteria, for
the month, place a 1 here.
   Acceptance Criteria:

   $\geq$ 80% True Positive
   $\leq$ 20% False Negatives
   $\leq$ 10% False Positive

Cum Avg. - the this is the average of the % TP, FP and FN , averaged
throughout the year beginning with January. For example, April's Cum. Avg will
be the average of the TP, FP and FN % of Jan, Feb, March and April. Average
each QC type.

QC / 1876b		#	PASS / FAIL

The 1876b spreadsheet is not included in the QC program package. It is a stand alone spreadsheet that performs the acceptance criteria calculations. Transfer the data to these boxes in the appropriate month. Do not complete is no analysis was performed in that month. (Reminder: 1876b is analyzed annually – at a minimum)

QC / INTER-LAB	Samples	Avg. Variance	# Out	% Out	Cum. Avg.

Samples – number of QC samples analyzed by an other laboratory.

Avg. Variance – this is the average of the variance found in the 'Original' column in the spreadsheet , TEM QC Summary. This information gives the overall precision of the laboratory for the month.
Note: use the true (and not absolute value) when averaging these numbers, as this will monitor any real bias a laboratory.

 # Out – number of outliers counted from the TEM QC Summary. (see also 2 and 3 in introduction)

% Out – The % of outliers to total QC samples

Cum. Avg. - this is the average of the variance, averaged throughout the year beginning with January. For example, April's Cum. Avg will be the average of the avg variance of Jan, Feb, March and April

QC / BLANKS	# Blanks	In Compliance?	# Out	% Out	Cum. Avg.

The tracking and evaluation of laboratory blank analysis is done by the laboratory manager. A reminder that the acceptance criteria for the blanks are:

the maximum allowable contamination levels for laboratory blanks are a cumulative average of 18 structures/mm^2 or any single preparation level of 53 structures/mm^2. Detailed information can be located in the appropriate SOP.

The manager should be recording blank analysis data and checking the data against the above criteria.

Note: this does not include field blank analysis.

Overall TEM Laboratory Performance	Total Samples	# Out	% Out	Cum. Avg.

This section is designed to demonstrate the laboratories overall performance by tracking the % of outliers against total samples analyzed. It does not provide a

summary for the analysts or laboratories precision (variance). This information is used to complete the

4.0 RESPONSIBILITY / REPORT REVIEW

This section describes the process of evaluation and review of the Monthly Quality Control Report. It delineates the responsibility to the applicable departments and defines the level of examination and reporting.

4.1 Laboratory Staff

The laboratory Management administrates the Monthly Quality Control Program at the local laboratory level. The Management is responsible for insuring compliance with the program requirements. The implementation of the program includes:

- Performance of the required amount of QC (listed on Summary Report, QA manual and section 2 above)
- Completion of the QC Summary Report
- Submittal of report to the QA Department on or before the 15th of the month following the month of report.
- Proper filing and maintenance of both the hard and electronic copy of the report

4.2 Quality Assurance Department

Review
It is the responsibility of the Corporate Quality Assurance Department to closely review and evaluate the laboratories monthly QC Report. The reports are reviewed and scored on:

- timeliness of submittal
- % QC of Sample totals
- coefficient of Variation determined (for PCM analysis)
- analysis of Standards

- calibration frequencies
- corrective actions
- instrument calibrations preformed at required frequencies
- within acceptance criteria

A checklist guides the reviewer through this evaluation process.

Performance Criteria

The QA Manager reviews the data for compliance with established acceptance criteria (see above analytical sections for specific standards). The data is compared to these standards. If the QC results and the calibration measurements are within control, a note is made on the checklist. No further action is required.

In the event of an outlier, an investigation is performed as to the cause. If the outlier is related to sample QC analysis, the QA Manager checks the corrective action report. The manager insures the actions taken have satisfied the performance criteria policies. A follow up with the Laboratory Manager may also be necessary.

If the laboratory shows non-compliance of calibration measurements, the QA Manager reviews the corrective action report. The issue may be discussed with the laboratory manager to confer on the severity of the problem, insure the instrument is currently in compliance, and provide additional support if necessary.

Reporting
The QA Department provides the laboratory manager feedback with a 'report card' on a semi annual basis. This report scores the laboratory performance on the checklist items listed above.

The QA Manager also issues periodic reports to the Corporate General Manager and/or President. These reports contain a summary of the laboratories performance as they relate to a QC activities.

The program (in sections or entirety) is assessed periodically for accuracy, applicability, efficiency. Any changes to the program must be approved by the QA Department.

Note:
It is not the intention of the Corporate QA Department to mandate the process by which QC data is collected. The day -to –day processes of the QC analysis is managed by the Laboratory Manager. For example, the Manager may choose to pull every 10th sample in a batch for QC analysis, or may choose to pull 10% at

the end of the work day. In other words, the procedures applied for the collection of data is done at the local level.

References:

New York State Department of Health, Environmental Laboratory Approval Program Certification Program. Item No.236, March 1, 1997.

AHERA, 1987. 40 CFR 763

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Method for The Determination of Asbestos In Bulk Building Materials, EPA/600/R-93-116

American Industrial Hygiene Association: *Quality Assurance Manual for Industrial Hygiene Chemistry*. Fairfax, VA. American Industrial Hygiene Association 1995

Appendix 4

- EMSL Analytical S.O.P. for AHERA Analysis, 40CFR Part 763 Appendix A to Subpart E Interim Transmission Electron Microscopy Analytical Methods, 7/02

EMSL Analytical S.O.P. for AHERA ANALYSIS

40CFR Part 763 Appendix A to Subpart E
Interim Transmission Electron Microscopy Analytical Methods

SAMPLE RECEIVING

Upon receipt of samples, check that the sample information on the Chain of Custody (COC) matches the information on the samples and other paperwork. Any discrepancies must be resolved before proceeding. If the samples do not have a COC then one is completed at time of log in. Have the client fill out the necessary information completely.

Information Required:
- Client name, address, telephone number, contact person, etc.
- Project number and state where samples were taken
- Number of samples sent and there ID
- Type of analysis requested
- Sample volumes or areas, where applicable
- Turn around time needed
- Date and time of delivery
- Date and signature of the person relinquishing the samples.
- Date and signature of the EMSL employee receiving the sample.

Check to see if the samples match the COC and if the cassettes are open, damaged, or contaminated.

If the samples are damaged or if the COC does not match, notify the client.

Clock in the samples and place your initials next to the time received.

If any of the following information is not supplied, contact the client.
- Client name, address, telephone number, contact person, fax number
- Project number/ name, state where samples where taken
- Number of samples sent and sample ID's
- Type of analysis requested
- Sample volumes or areas if applicable
- Turn around time-"RUSH" is not acceptable
- A date and signature of the person relinquishing the samples
- All samples MUST be accounted for with the proper sample ID's
- All samples MUST be sealed, properly bagged and undamaged

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SAMPLE LOG IN

If all of the above criteria for sample receiving are met then the sample can be logged in to Sample Master (LIMS) as per the Sample Master SOP. This process will assign a unique EMSL order number for the project as well as unique lab sample ID's. The LIMS will create:

- appropriate bench sheets for the type of analysis requested
- Internal Chain of Custody

SAMPLE PREPARATION

Almost all AHERA samples are collected on MCE filters (0.45 micron or less pore size). Polycarbonate (PC) filters are also acceptable (0.4 micron or less pore size).

MCE Filters

1. Place the samples in an order corresponding to the clients COC.
2. Cut a wedge from the filter of each sample and place it on a clean 1 x 3 microscope slide in the following manner.
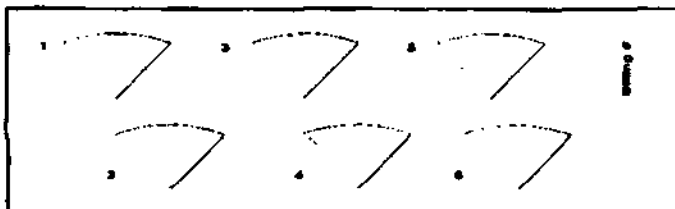
(Note: it is also helpful to cut your lab blank a slightly different shape wedge to aid in differentiation between the first and last sample. In this example, 1 and 6 represents the client sample number)

3. Add a Laboratory Blank to the end of the series. Only done on a daily basis.
4. Collapse the filters using the acetone vaporizer and fresh acetone.
5. Write the order billing number and the first and last client sample number for the sample set, with a permanent marker (sharpie).
6. Ash the sample for a 5% ashing. The current calibrated time for a 5% ash should be posted on the asher unit itself. See Asher SOP for instructions on its use.
7. When ashing is done, remove sample(s) from the asher.
8. Transfer the slide(s) of samples from the asher to the vacuum evaporator for carbon coating. (See Vacuum Evaporator SOP for details in use).
9. Coat the samples with a carbon coat that is brownish/gray. Too little carbon and you will end up with a lot of breakage. Too much carbon (mirrored surface) and you will see cracking (fine fault lines) at analysis.
10. After carbon coating, the samples are ready for filter dissolution in the Jaffe wick washers.

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11. Place a piece of cut Kimwipe onto the sponge in the petri dish. Add enough acetone to completely saturate the sponge and Kimwipe, bringing the final volume to half the thickness of the sponge. It is suggested to cut a notch in the top right corner of the Kimwipe as a visual aid in orientation. (Optional) DMF and DMSO may replace acetone and in some cases will yield better preparation.

12. Place three grids per sample in order on top of the Kimwipe making sure that the "matte" or dull side of the grid is up.

13. With a straight edge razor blade cut a thin strip (5-6 mm) from the rounded edge to the print of the collapsed and sated sample fiber wedges.

14. Again with the straight edge razor blade gently life up a corner of the thin strip to aid in removal off the slide with forceps.

15. Using clean forceps, lift up one strip at a time and place it onto one row of the copper grids, carbon side up

Set up as shown:

Sample # ——→ 1   2   3   4   5
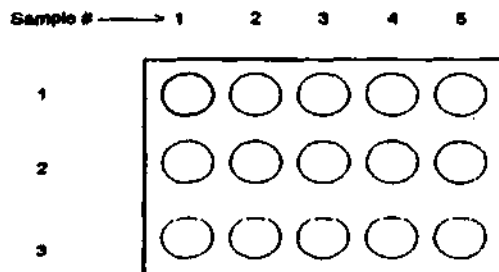
16. Once all strips have been placed on the grids, add acetone if needed to remove any air bubbles under the filter paper.

17. Replace the petri lid and label.

18. Allow to stand for 35 to 45 minutes.

19. Remove the petri lid. With fine forceps, pick up the kimwipe by a corner and remove it (and the samples) from the wick washer. Place kimwipe onto a new kimwipe or a paper towel with grids facing upward.

20. Pull each grid carefully off the kimwipe with fine forceps.

21. Grids are attached to an asymmatric copper clip with pre-cut sections of carbon double-stick tape. The clips are rectangular with a rectangular elongate opening running along the long axis of the clip. One end has a semi-circular notch on one end.

22. Counting away from the notch, the first and second grids are from sample one, the third and fourth from the second sample, and the fifth and sixth from the third sample. On the second clip are samples, four in the first and second grid positions, five in the third and fourth positions and the lab blank positions five and six. In the event of additional samples, continue this sequence, placing the blank at the appropriate position.
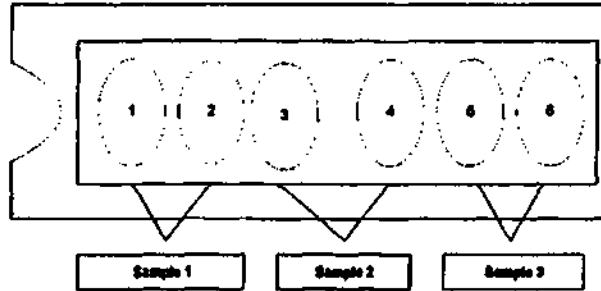
23. The notched end of the clip is oriented closest to the tip of the specimen arm.

Grid Storage

1. Clips are lettered A through U inclusive, and placed in a specially designed holding box.
2. Grid boxes are uniquely and sequentially numbered (eg. NY0217)
3. The analytical worksheet is labeled with a grid box number, clip letter (ID) and location #. The location number 1-6 starting from the notch.
4. All unanalyzed grids are placed into standard numbered grid boxes, recorded on the grid box log sheet and stored for 3 years.

NOTE: Polycarbonate (PC) Filters (0.4 microns or less pore size)

It is unlikely that you will ever receive AHERA samples on PC filters, however, if you do, the preparation steps are identical EXCEPT that the collapsing and ashing steps (6-8 above) are not performed. In addition, as acetone will not dissolve polycarbonate. Chloroform is used in the Jaffe wick washer instead.

SAMPLE ANALYSIS

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Procedures

1. Remove the first sample grid from the box and insert it into the TEM.
2. Bring the TEM to a magnification of 300 to 500x and inspect the grids to determine if at least 50% if the grid openings are intact. If two of the three grids are not 50% intact then the samples will have to be reprepped.
3. Two grids are analyzed per sample. (Separation of grid openings for each grid is recorded on the bench sheet, indicating the column location of the particular grid).

Sample Preparation Acceptance

1. More than 50% of the grid must be covered by the replica.
2. Grids must have at least 50% intact grid openings.
3. Grids must not have more than 10% opaque area due to incomplete filter dissolution.
4. Total Grid openings must have <50% overlapping of folded replica film
5. At least 20 grid openings with <5% holes and <5% opaque area due to incomplete filter dissolution.
6. Grid openings analyzed must not have rips or overlapping folds.

AHERA Protocol

1. At a magnification of 100X, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 19,000x taking care to remain in the chosen grid opening.
3. Log the grid opening identification on the sample worksheet.
4. Move to the upper left corner of the grid square and begin traversing the grid towards the lower left corner (using only one directional control). Once the opposite grid bar has been reached, move over in the right direction, one large circle (~5 µm) width and proceed scanning in the upwards direction. Repeat procedure. The entire grid opening has been scanned. Take care not to count any structure twice or to miss any area of the grid opening.
5. If no fibrous structures have been located repeat steps one through four until the stopping procedures have been reached. [Do not analyze adjacent grid openings].
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis. See Energy Dispersive X-Ray Analysis SOP.
7. Obtain a diffraction pattern for the sample. Record diffraction information on the sample worksheet. See SAED SOP for detailed information on obtaining diffraction patterns. If diffraction pattern is consistent with asbestos proceed to EDXA
8. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet.
9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.

10. For asbestos structures, note whether it is a fiber, bundle, cluster, or matrix.
- *Fiber:* a single fibril not connected to any non-fibrous debris.
- *Bundle:* any group of three or more fibers lying parallel and with less than a single fiber diameter between them.
- *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
- *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
11. Working magnification is 19,000x to 20,000x.
12. Fiber criteria
- Aspect ratio of 5:1
- Must be $\geq 0.5\mu m$ length
13. Sizing of fibers
- Record $<5\mu m$ length and $\geq 5\mu m$ length
14. Required EDX (for each type of asbestos)
- All fibers that cause the sample to exceed $70str/mm^2$
15. Required diffraction patterns (for each type of asbestos)
- All fibers that cause the sample to exceed $70str/mm^2$
- One micrograph per 5 samples that contain asbestos
16. Stopping rules
- Count the number of grids required to reach a detection limit of <0.005
- Analyst may stop if 50 fibers in a minimum of 4 grid openings is reached
17. Required detection limit
- 0.005 fibers/cc
18. Laboratory blanks
- One blank per sample set
19. Required filters
- $0.45\mu m$ MCE
20. Pass / Fail limit
- Average of inside samples $<70str/mm^2$
- Pass the "Z" test (Optional)

21. Sample Requirements
- Requires a volume of 1200 to 1800 liters in most instances
- Requires Inside, Outside, and Blank samples

If these requirements have not been met, a disclaimer must appear on the report.

EQUATIONS   CONFIDENTIAL

Air Samples

EFA = Effective filter area of a 25mm cassette = 385mm^2
GOA = Grid opening area (0.00635)
N = Number of fibers (If N=0 then default to 1 structure)
V = Volume (1200 liters)
AA= Area analyzed (0.06985)
NO = Number of openings analyzed (11)
n = Number of Samples

Area Analyzed = (GOA x NO)   [0.06985mm^2 = (0.00635 x 11)]

Structures /mm^2 = (N / AA)   [14.3/mm^2 = (1 / 0.06985)]

Structures /cc = $\dfrac{385 \times N}{(V \times AA \times 1000)}$ $\dfrac{385 \times 1}{[0.0046/cc = (1200 \times 0.06985 \times 1000)]}$

# of openings to read for 0.005^s/cc sensitivity

$$NO = (EFA \times 10^{-3}) / (GOA \times V \times 0.005)$$

Round all decimals UP to the next higher whole number :
          10.1 (11) = (385) / (1200 x 0.00635 x 10^3 x 0.005)

Z" Test

$\underline{Y_i}$ = the mean of the natural logarithms of the insides (str/cc)   Y = ln N1 + ln N2 + ln

N3 + ln N4 + ln N5
$\overline{Y_o}$ = the mean of the natural logarithms of the outsides (str/cc)   n

Assume the insides to be... .06   the outsides to be... .0062
                             .041   .0031
                             .063   .0032
                             .046   .0030
                             .036   .0034

"Z" Test = $\dfrac{\overline{Y_i} - \overline{Y_o}}{0.8 (1/n_i + 1/n_o)^{0.5}}$ 5.11 = $\dfrac{(-3.04) - (-5.62)}{0.8 (0.4)^{1/2}}$

The Site passes if Z < 1.65

SAFETY

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Asbestos:
Prudent measures must be taken to prevent any possible airborne asbestos fiber release from occurring during sample handling. Any filter handling preformed prior to the acetone collapse step should be performed under the safety hood.

Acetone:
Keep away from heat, sparks, flame. Avoid breathing vapors - use with adequate ventilation. Avoid contact with eyes. Prevent prolonged or repeated contact with skin.

Carbon Spark:
Shield eyes from the glow of the spark during the carbon coating process.

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Signature Page

In signing this, I acknowledge having read and understood the previous pages of this document.

APPROVED BY:

LABORATORY Manager(print)	LABORATORY Manager (sign)	DATE

READ AND UNDERSTOOD BY:

Print Name	Signature	Date
1)		
2)		
3)		
4)		
5)		
6)		
7)		
8)		
9)		
10)		

Appendix 5

- EMSL Analytical, Inc. - Standard Operating Procedures, Asbestos
  Analysis, Phase Contrast Microscopy (PCM), 8/1/00

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Standard Operating Procedure
for Phase Contrast Microscopy (PCM)

EMSL Analytical, Inc.

Standard Operating Procedures
Asbestos Analysis
Phase Contrast Microscopy (PCM)

Revision Date: July 2000
Original Date: June 1995

Issue Date: August 1, 2000

EMSL Analytical Inc., Quality Assurance Dept.

Table of Contents

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1.0 SAMPLE RECEIVING

1. Upon receipt of samples, check that the sample information on the Chain of Custody matches the information on the samples and any other paperwork. Any discrepancies must be dealt with before proceeding. If the samples do not have a COC, then provide a blank one, having the client fill out the necessary information.
    Information includes but is not limited to:
    - Client name, address, telephone number, fax #, contact person, etc.
    - State of origin
    - Number of samples sent and their ID
    - Type of analysis requested
    - Sample volumes or areas, where applicable
    - Turn around time
    - Signature, date, and time of the person relinquishing the samples.
    - Signature, date, and time of an EMSL employee receiving the sample.

2. Acceptability of the condition of the samples is acknowledged upon signing the chain of custody.
    Insure samples comply with the following criteria:
    - Correct methodology requested
    - Properly labeled with unique identification information
    - Samples are not packaged using expanded Styrofoam type pieces
    - Samples are submitted separately from bulk asbestos samples
    - Correct sampling media is used

3. Log samples into LIMS system with all appropriate data:
    a. Computer assigns unique, sequential billing/job numbers, and unique sequential individual sample numbers.
    b. Sample batches are labeled with billing number, due date and time.
    c. Worksheets and billing sheets are printed and placed with sample batch

4. Samples with chain of custody, work sheet, billing worksheet, and all other associated paperwork are taken to the air sample preparation station.

2.0   SAMPLE PREPARATION

MCE FILTERS

1. Place the samples in an order corresponding to the clients COC.

2. On a pre-cleaned 1X3-microscope slide oriented in a horizontal position, using a permanent marker, write the billing and sample number on the slide.

3. Using a number ten scalpel blade with a rocking motion, cut a wedge from the filter and place it on the slide taking care to handle the filter only by the unexposed edge.

4. Collapse the filters using the acetone vapor generator by injecting acetone into the vapor generator with a syringe.

5. With permanent marker (such as a 'Sharpie'), outline the collapsed filter on the underside of the slide.

6. Place a drop of triacetin on the filter and cover with a clean cover slip.

3.0   MICROSCOPE CALIBRATION

1.   Turn on the microscope.

2.   Slowly increase the power to the lamp.

3.   Adjust the light source for even illumination across the entire sample.

4.   Insert the telescope ocular into the head of the microscope in place of the 10x ocular.

5.   Focus the image of rings in the ocular.

6.   If the rings do not form concentric circles adjust them using the adjustment screws on the condenser.

7.   Record the date and alignment in the microscope logbook.

8.   Make sure the field iris is in focus, centered on the sample and open only enough to fully illuminate the field of view.

9.   (Weekly) Check the phase-shift detection limit using the phase-contrast slide.
   - Center the HSE/NPL phase-contrast slide to focus under the PCM.
   - The lines in blocks 1 through 3 must be fully visible showing row 4 & 5 as partially visible and the least distinct.
   - Rows 6 and 7 must be invisible. Record results in logbook.

8.   (Monthly) Check measurement of the Walton Beckett graticle. Using a stage micrometer, determine the diameter of the graticle circle. Acceptable values are 100 microns +/- 2 microns. Calculate area of graticule using πR^2 where:

$$\pi = 3.14$$
$$R = radius \ (mm)$$

The calculated value should be 0.00785 mm^2 for a graticle measuring 100 µm.

9.   If image quality is poor, clean the microscope optics or inform the supervisor.

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4.0   SAMPLE ANALYSIS

1. Fill out the appropriate information on the provided PCM bench sheet.

2. Place the slide containing the first sample onto the stage and center it under the objective lens. *Make sure to use the 40x objective.*

3. Focus the sample taking care not to allow the objective lens to touch the slide.

4. Starting at the upper left corner of the sample, traverse randomly down the filter in increments large enough to avoid possible overlapping of the fields of analysis.

5. If more than one-sixth of the first twenty fields of view is covered with particulate, analysis must be discontinued and the sample declared overloaded.

6. Once stopped on a field of analysis slowly over and under focus to see any fibers that might be imbedded in the filter medium.

7. Working magnification is 400x.

8. Fiber criteria
    - Aspect ratio of 3:1
    - Must be ≥ 5 micron in length

9. Count those fibers, which are within the boundaries of the circular graticule.

10. For fibers crossing the boundaries of the graticule
    - Count as a half fiber any fiber with only one end lying within the graticule field.
    - Do not count any fiber, which crosses the boundary more than once.
    - Count bundles of fibers as one fiber unless observing both ends of a fiber can identify individual fibers.

11. Sizing of fibers
    - Count if ≥ 5 micron in length with 3:1 aspect ratio
    - ≥ 5 micron in length with 3:1 aspect ratio protruding from a matrix material

12. Stopping rules
    - Count 100 graticule fields or 100 fibers whichever comes first.
    - Count a minimum of 20 fields regardless.
    - Complete count of final field; do not terminate count mid-field.

13. **Blanks**
    - Prep and analyze all blanks
    - Subtract the average of the blanks from the sample result for the associated batch

14. **Required filters**
    - 0.45 – 1.2 micron pore size, 25mm mixed cellulose ester (MCE)

15. **Pass / Fail limit**
    - Depends on job requirements

5.0   CALCULATIONS

Results are reported in units of fibers/cc if sampling information is provided. In the event sample volume information is not submitted, report results in concentrations of fibers/mm^2.

To calculate fiber density in fibers/mm^2 :
- Total fibers counted minus mean field blank count = fibers
- Fibers / # fields counted x area of graticule = f/mm^2

To calculate fibers/cc:
- Fibers/filter = f/mm^2 x effective field area of sampling cassette (385mm for 25mm)

- $$f/cc = \frac{fibers/filter}{sample\ volume\ (l)\ X\ 1,000}$$

Similarly
- Fibers/mm^2 (E) = (F/n$_f$ - B/n$_b$) / A$_f$
- Fibers/cc (C) = (E)(A$_c$) / V x 10^3

Where:

E	= fiber density in fibers/mm^2
F/n$_f$	= average fiber count per graticule field
B/n$_b$	= mean field blank count per graticule field
A$_f$	= graticule field area (approx. 0.00785 mm^2)
A$_c$	= effective collection area of filter (approx. 385 mm^2 for 25 mm cassette)
V	= volume in liters

EXAMPLE

Fibers = 35
Mean blank value = 0
Fields = 53
Volume = 1200 liters
Cassette size = 25mm (effective field area of 385mm)

f/mm^2 :   35.0 fibers/(53 fields x .00785) = 84.0 f/mm^2

Fibers /cc = $\dfrac{84.0 \times 385}{(1200 \times 1000)}$ =   0.027 f/cc

6.0   REPORTING RESULTS

<u>Data is reported in the final report and includes</u>:

- Identification of laboratory location
- Page number
- Name and address of client
- Unique ID of test report
- Name and address of client
- Test method (NIOSH 7400 Issue 2, Fourth Edition August 15, 1994)
- Sample ID#, location, sample date, volume, fibers counted, fields counted, and limit of detection.
- Concentrations f/mm^2 and f/cc

<u>Disclaimers cited in each report are as follows</u>:
"The laboratory is not responsible for data reported in fibers/cc, which is dependent on volume collected by non-laboratory personnel."

"This report relates only to the samples reported above. This report may not be reproduced, except in full, without written approval by EMSL"

<u>Limit of Detection</u>
The recognized method detection limit for NIOSH 7400 is 5.5 fibers counted in 100 fields. Concentrations below 5.5 fibers are reported as less than (<) 5.5 fibers in 100 fields, or a fiber density of <7 fibers/mm^2.

7.0   QUALITY CONTROL

Procedures

1. After daily microscope calibration and before beginning analysis, analyze one reference slide.
    - Record results
    - If results are acceptable, proceed with analysis of samples.

2. Analyze all field blanks that have come with the sample set.
    - If the blanks contain >7 fibers in 100 fields then report possible contamination to the client.

3. Analyze a blind recount for every ten samples analyzed, record results in QC logbook.

4. All analysts participate in a Round Robin and PAT program.

5. New analyst proficiency is determined through evaluation of their skills using a training log.

QC Data Management

Accuracy

The analysis of reference slides (old proficiency test samples with known concentrations) are analyzed daily by each analyst. This analysis is performed prior to analysis of client samples. This data is checked against acceptable limits as determined by the issuing agency.

Precision

Daily reference sample data for each analyst is collected and tracked in the EMSL Monthly QC report. For each set of 20 data points, a coefficient of variation (CV) is determined for each individual analyst in fiber ranges of:

> 5-20 fibers/100fields
> 21-50 fibers/100 fields
> 51-100 fibers/100 fields

As these standard samples are analyzed, current data replaces the oldest data point.

Sample reanalysis is performed as intra analyst (same analysts) QC at the rate of 10% of sample volume. This data is tracked and managed in the EMSL Monthly QC report. The pass/fail criteria follows NIOSH 7400 requirements as follows:

Pass - If the absolute value of the difference between original and QC analysis (in f/mm2) is less than or equal to the value calculated by the constant (2.77) multiplied by the average of the square root of the original and QC fiber counts times the calculated CV value divided by 2.

$$\left| \sqrt{original f \ / \ mm^2} - \sqrt{qcf \ / \ mm^2} \right| \leq 2.77 \left(\frac{\sqrt{f \ / \ mm^2} + \sqrt{f \ / \ mm^2}}{2} \right) \frac{CV}{2}$$

Fail - If the absolute value of the difference between original and QC analysis (in f/mm2) is greater than the value calculated by the constant (2.77) multiplied by the average of the square root of the original and QC fiber counts times the calculated CV value divided by 2.

$$\left| \sqrt{original f \ / \ mm^2} - \sqrt{qcf \ / \ mm^2} \right| > 2.77 \left(\frac{\sqrt{f \ / \ mm^2} + \sqrt{f \ / \ mm^2}}{2} \right) \frac{CV}{2}$$

References:

NIOSH Manual of Analytical Methods (NMAM) 7400 Issue 2:August 15, 1994. Fourth Edition

American Industrial Hygiene Association: *Quality Assurance Manual for Industrial Hygiene Chemistry*. Fairfax, VA. American Industrial Hygiene Association 1995

Appendix 6

- EMSL Analytical, Inc. – NIST Bulk Asbestos Proficiency Test, April 2003, Round M12003

- EMSL Analytical, Inc. – United States Department of Commerce National Institute of Standards and Technology, NVLAP, Certificate of Accreditation, EMSL Analytical Inc. Mobile Laboratory, Airborne Asbestos Fiber Analysis, June 30, 2004

- EMSL Analytical, Inc. – United States Department of Commerce National Institute of Standards and Technology, NVLAP, Certificate of Accreditation, EMSL Analytical Inc. Mobile Laboratory, Bulk Asbestos Fiber Analysis, June 30, 2004

- Department of Public Health and Human Services Environmental Laboratory, State of Montana, Certification for Drinking Water Analysis, Expiration Date: 9/18/06

- EMSL Analytical, Inc., Outline of the Laboratory Quality Assurance Program, Phase Contrast Microscopy Transmission Electron Microscopy Polarized Light Microscopy, 12/03

July 22, 2003

Mr. Robert DeMalo
EMSL Analytical Inc.
107 Haddon Avenue
Westmont, NJ 08108-2799

NVLAP Lab Code: 200481 - 0

Dear Mr. DeMalo,

Congratulations! Your laboratory has passed the April 2003 round of proficiency testing
(PLMM12003) required by the National Voluntary Laboratory Accreditation Program
(NVLAP) for Bulk Asbestos Analysis.

If your laboratory is accredited, your status remains unchanged. If your laboratory is not
yet accredited, or if your laboratory's accreditation has been suspended, you will be
notified of any requirements your laboratory must meet to complete the accreditation /
reaccreditation process.

Enclosed you will find the Summary of Analysis and your laboratory's results.

If you have any questions, please call Thomas R. Davis at 301-975-6499, or Hazel M.
Richmond at 301-975-3024.

Sincerely,

Warren R. Merkel, Chief
Laboratory Accreditation Program

Enclosure(s)

NIST

NVLAP Lab Code 200481-0

PROFICIENCY TEST M12003 SUBTOTALS

Sample 1   0

Sample 2   0

Sample 3   0

Sample 4   0

TOTAL POINTS   0

Failure = 150 or more total points

NVLAP Lab Code 200481-0

SAMPLE 1

Criteria	Reported by Laboratory	Reference Values	Acceptable Answers	Assigned Points
Asbestos Type (150 pts./type)	NONE	None	None	0

Total Points Assigned for Sample 1 = 0

NVLAP Lab Code 200481-0

SAMPLE 2
Asbestos Type I

Criteria	Reported by Laboratory	Reference Values	Acceptable Answers	Assigned Points
Asbestos Type (150 pts./type)	AMOS	Amosite	Amosite	0
Reporting Additional Asbestos Type (150 pts. if >0.1%, 75 pts. if 0.1%)	NONE	None	None	0
% Asbestos (50 pts.)	15.0	9.9	2.0 to 20.0	0
Color (10 pts.)	CL	CL (colorless)/ GR (green)/ OT (other)	CL (colorless)/ GR (green)/ OT (other)	0
Pleochroism (10 pts.)	N	N (none)	N (none)	0
Extinction (10 pts.)	P	P (parallel)	P (parallel)	0
Sign of Elongation (10 pts.)	P	P (positive)	P (positive)	0
Average Refractive Index (40 pts. each index, 10 pts if $\gamma=\alpha$ or γ & α reversed)	1.700 1.681	$\gamma=1.700$ $\alpha=1.679$	$\gamma=1.691$ to 1.709 $\alpha=1.670$ to 1.688	0
Birefringence (10 pts.)	M	M (medium)	M (medium)	0

NVLAP Lab Code 200481-0

SAMPLE 2
Asbestos Type 2

Criteria	Reported by Laboratory	Reference Values	Acceptable Answers	Assigned Points
Asbestos Type (150 pts./type)	CHRY	Chrysotile	Chrysotile	0
Reporting Additional Asbestos Type (150 pts. if >0.1%, 75 pts. if 0.1%)	NONE	None	None	0
% Asbestos (50 pts.)	7.0	1.1	0.1 to 10.0	0
Color (10 pts.)	CL	CL (colorless)	CL (colorless)	0
Pleochroism (10 pts.)	N	N (none)	N (none)	0
Extinction (10 pts.)	P	P (parallel)	P (parallel)	0
Sign of Elongation (10 pts.)	P	P (positive)	P (positive)	0
Average Refractive Index (40 pts. each index, 10 pts if $\gamma=\alpha$ or γ & α reversed)	1.554 1.551	$\gamma=1.559$ $\alpha=1.548$	$\gamma=1.552$ to 1.566 $\alpha=1.541$ to 1.555	0
Birefringence (10 pts.)	L	L (low) to M (medium)	L (low) to M (medium)	0

NVLAP Lab Code 200481-0

SAMPLE 2
Asbestos Type 3

Criteria	Reported by Laboratory	Reference Values	Acceptable Answers	Assigned Points
Asbestos Type (150 pts./type)	NONE	Crocidolite	Crocidolite/ None	0
Reporting Additional Asbestos Type (150 pts. if >0.1%, 75 pts. if 0.1%)	NONE	None	None	0
% Asbestos (50 pts.)	0.0	Trace	0 to 0.1	0
Color (10 pts.)		BL (blue)		0
Pleochroism (10 pts.)		Y (yes)		0
Extinction (10 pts.)		P (parallel)		0
Sign of Elongation (10 pts.)		N (negative)	Not Scored	0
Average Refractive Index (40 pts. each index, 10 pts if γ=α or γ & α reversed)		γ=1.705 α=1.691		0
Birefringence (10 pts.)		L (low) to M (medium)		0

Total Points Assigned for Sample 2 asbestos types 1-3 = 0

NVLAP Lab Code 200481-0

SAMPLE 3
Asbestos Type 1

Criteria	Reported by Laboratory	Reference Values	Acceptable Answers	Assigned Points
Asbestos Type (150 pts./type)	CROC	Crocidolite	Crocidolite	0
Reporting Additional Asbestos Type (150 pts. if >0.1%, 75 pts. if 0.1%)	NONE	None	None	0
% Asbestos (50 pts.)	15.0	7.8	2.0 to 15.0	0
Color (10 pts.)	BL	BL (blue)/ OT (other)	BL (blue)/ OT (other)	0
Pleochroism (10 pts.)	Y	Y (yes)	Y (yes)	0
Extinction (10 pts.)	P	P (parallel)	P (parallel)	0
Sign of Elongation (10 pts.)	N	N (negative)	N (negative)	0
Average Refractive Index (40 pts. each index, 10 pts if $\gamma=\alpha$ or γ & α reversed)	1.705 1.697	$\gamma=1.699$ $\alpha=1.688$	$\gamma=1.679$ to 1.719 $\alpha=1.668$ to 1.708	0
Birefringence (10 pts.)	L	L (low) to M (medium)	L (low) to M (medium)	0

NVLAP Lab Code 200481-0

SAMPLE 3
Asbestos Type 2

Criteria	Reported by Laboratory	Reference Values	Acceptable Answers	Assigned Points
Asbestos Type (150 pts./type)	CHRY	Chrysotile	Chrysotile	0
Reporting Additional Asbestos Type (150 pts. if >0.1%, 75 pts. if 0.1%)	NONE	None	None	0
% Asbestos (50 pts.)	7.0	2.4	0.1 to 10.0	0
Color (10 pts.)	CL	CL (colorless)	CL (colorless)	0
Pleochroism (10 pts.)	N	N (none)	N (none)	0
Extinction (10 pts.)	P	P (parallel)	P (parallel)	0
Sign of Elongation (10 pts.)	P	P (positive)	P (positive)	0
Average Refractive Index (40 pts. each index, 10 pts if $\gamma = \alpha$ or γ & α reversed)	1.554 1.552	$\gamma=1.559$ $\alpha=1.549$	$\gamma=1.552$ to 1.566 $\alpha=1.542$ to 1.556	0
Birefringence (10 pts.)	L	L (low) to M (medium)	L (low) to M (medium)	0

Total Points Assigned for Sample 3, asbestos types 1-2 = 0

NYLAP Lab Code 200481-0

SAMPLE 4

Criteria	Reported by Laboratory	Reference Values	Acceptable Answers	Assigned Points
Asbestos Type (150 pts./type)	CHRY	Chrysotile	Chrysotile	0
Reporting Additional Asbestos Type (150 pts. if >0.1%, 75 pts. if 0.1%)	NONE	None	None	0
% Asbestos (50 pts.)	2.0	3.1	0.1 to 10.0	0
Color (10 pts.)	CL	CL (colorless)	CL (colorless)	0
Pleochroism (10 pts.)	N	N (none)	N (none)	0
Extinction (10 pts.)	P	P (parallel)	P (parallel)	0
Sign of Elongation (10 pts.)	P	P (positive)	P (positive)	0
Average Refractive Index (40 pts. each index, 10 pts if γ=α or γ & α reversed)	1.554 1.551	γ=1.559 α=1.549	γ=1.552 to 1.566 α=1.542 to 1.556	0
Birefringence (10 pts.)	L	L (low) to M (medium)	L (low) to M (medium)	0

Total Points Assigned for Sample 4 =   0

158049

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
INDIVIDUAL LABORATORY REPORT FOR ROUND 133
LAB ID=158049   NOVEMBER 11, 2003
EMSL ANALYTICAL, INC., LIBBY, MT 59923

CONTAMINANT (ABV.)	UNIT	SAMPLE NO.	REPORTED RESULTS	MEAN VALUES *	ACCEPTABLE RANGE# LOWER	UPPER	Z & SCORE	LAB @ PERFORMANCE
ASBESTOS/FIBERS (ASB/NMF)	(F/MM2)	1	108.3000	125.6590	61.5729	212.3637	-0.72	A
	(F/MM2)	2	196.0000	239.6601	117.4286	405.0087	-0.96	A
	(F/MM2)	3	121.0000	160.7504	78.7579	271.6344	-1.32	A
	(F/MM2)	4	70.0000	78.3428	38.3880	132.3996	-0.55	A

* Mean values are the mean of all laboratories based on original scales except for asbestos.
  Asbestos results are calculated based on transformed data. Therefore, asbestos performance limits are
  not symmetrical to the mean values.
Upper limit: mean value + 3 standard deviations
  Lower limit: mean value - 3 standard deviations
& Z Score = (reported result-mean value)/standard deviation
@ A: Analysis acceptable   -: Results not reported
  H: Results > upper limit (Z > 3), not acceptable   L: Results < lower limit (Z < -3), not acceptable
Note: the acceptability of reported results is based on z-scores. This is why a reported result may appear
      acceptable according to performance limits, but be identified as an outlier.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
LABORATORY YEAR-TO-DATE PERFORMANCE REPORT FOR ROUND 155
LAB ID=156049 NOVEMBER 11, 2003
EMSL ANALYTICAL, INC., LIBBY, MT 59923

SAMPLE TYPE	ROUND	ROUND * PERFORMANCE	ACCUMULATED PERFORMANCE		PROFICIENCY RATING #
			4 ROUNDS (%)	2 ROUNDS (%)	
ASBESTOS/FIBERS	153	3/4			
	154	4/4			
	155	4/4	11/12 92	8/8 100	P

* The denominators represent the total number of samples to be analyzed.
  The numerators represent the number of acceptable results.
  A '-' represents non-submittal and is calculated as a zero in the numerator.
P : Proficient   N: Nonproficient
  Performance ratings are based on expected results over four rounds (one year).   A lab's
  performance on each sample type is rated proficient (P), if: 1) three-fourths   (75%) or more
  of the accumulated results over four rounds are acceptable or 2) for the   last two rounds,
  all samples are analyzed and the results are 100% acceptable. If a   laboratory receives
  samples for a contaminant and does not report the data, the results will be   considered
  unacceptable for that contaminant.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
SUMMARY RESULTS OF ALL LABS FOR ROUND 155

CONTAMINANT (UNIT)	SAMPLE NO.	SUMMARY STATISTICS OF ALL LABS				# LABS RATED	# OF LABS ACCEPTABLE	# LOW OUTLIER	# HIGH OUTLIER
		N	MEAN*	STD@	RSD(%)&				
CADMIUM (MG)	1	229	0.0089	0.000168	4.3	220	213	10	6
	2	229	0.0116	0.000465	4.0	229	219	7	3
	3	229	0.0154	0.000618	4.0	229	216	9	4
	4	229	0.0077	0.000307	4.0	229	215	9	5
LEAD (MG)	1	232	0.0576	0.003505	4.0	232	223	4	5
	2	232	0.0294	0.001177	4.0	232	213	10	9
	3	232	0.0585	0.002339	4.0	232	220	7	5
	4	232	0.1105	0.00466	4.0	232	218	7	7
ZINC (MG)	1	229	0.1002	0.004074	4.1	229	216	7	6
	2	229	0.1959	0.007836	4.0	229	215	10	6
	3	229	0.0499	0.002144	4.3	229	208	10	11
	4	229	0.1475	0.0059	4.0	229	207	12	10
SILICA (MG)	1	67	0.0503	0.008839	17.6	67	63	1	3
	2	67	0.0995	0.015089	15.2	67	64	2	1
	3	67	0.1083	0.016189	15.0	67	66	1	0
	4	67	0.0701	0.011388	16.2	67	65	0	2
ASBESTOS/FIBERS (F/MM2) +	1	166	125.6590	25.1318	20.0	842	696	74	72
	2	164	239.6601	47.93002	20.0	842	693	120	29
	3	164	160.7304	32.14608	20.0	842	720	89	33
	4	164	78.3428	15.66857	20.0	842	773	27	42
CHLOROFORM (MG)	1	196	0.2995	0.012528	4.1	196	182	7	7
	2	196	0.4942	0.01977	4.0	196	183	5	8
	3	196	1.1335	0.045341	4.0	196	182	6	8
	4	196	0.1013	0.005991	5.9	196	181	6	9
1,2-DICHLOROETHANE (MG)	1	196	1.2324	0.049297	4.0	196	186	6	4
	2	196	0.9182	0.036729	4.0	196	190	3	3
	3	196	0.3915	0.019661	4.0	196	188	2	6
	4	196	0.2050	0.008324	4.1	196	183	5	8
TRICHLOROETHYLENE (MG)	1	197	0.8776	0.035106	4.0	197	189	4	4
	2	197	1.1292	0.045168	4.0	197	186	7	4
	3	197	0.2965	0.011651	4.0	197	183	7	7
	4	197	0.4888	0.019553	4.0	197	185	6	6

* MEAN - The mean of all laboratories. These values were
         listed on the individual laboratory report.
@ STD - standard deviation
& RSD - relative standard deviation=((STD/MEAN)*100%)
+   - Results for fibers are calculated on transformed data.

United States Department of Commerce
National Institute of Standards and Technology

NVLAP ®

ISO/IEC 17025:1999
ISO 9002:1994

Certificate of Accreditation

EMSL ANALYTICAL INC. MOBILE LABORATORY
WESTMONT, NJ

is recognized by the National Voluntary Laboratory Accreditation Program
for satisfactory compliance with criteria set forth in NIST Handbook 150:2001,
all requirements of ISO/IEC 17025:1999, and relevant requirements of ISO 9002:1994.
Accreditation is awarded for specific services, listed on the Scope of Accreditation, for:

AIRBORNE ASBESTOS FIBER ANALYSIS

June 30, 2004

Effective through

For the National Institute of Standards and Technology

NVLAP Lab Code: 200481-0

NVLAP-C1C (06-01)

NVLAP®

National Institute of Standards and Technology

National Voluntary Laboratory Accreditation Program

ISO/IEC 17025:1999
ISO 9002:1994

Scope of Accreditation

AIRBORNE ASBESTOS FIBER ANALYSIS   NVLAP LAB CODE   200481-0

EMSL ANALYTICAL INC. MOBILE LABORATORY
107 Haddon Avenue
Westmont, NJ 08108-2799
Mr. Robert DeMalo
Phone: 856-858-4800   Fax: 856-858-1292
E-Mail: rdemalo@EMSL.com

NVLAP Code	*Designation*
18/A02	U.S. EPA's "Interim Transmission Electron Microscopy Analytical Methods-Mandatory and Nonmandatory-and Mandatory Section to Determine Completion of Response Actions" as found in 40 CFR, Part 763, Subpart E, Appendix A.

June 30, 2004

Effective through   For the National Institute of Standards and Technology

NVLAP-01S (06-01)

United States Department of Commerce
National Institute of Standards and Technology

NVLAP ®

ISO/IEC 17025:1999
ISO 9002:1994

Certificate of Accreditation

EMSL ANALYTICAL INC. MOBILE LABORATORY
WESTMONT, NJ

is recognized by the National Voluntary Laboratory Accreditation Program
for satisfactory compliance with criteria set forth in NIST Handbook 150:2001,
all requirements of ISO/IEC 17025:1999, and relevant requirements of ISO 9002:1994.
Accreditation is awarded for specific services, listed on the Scope of Accreditation, for:

BULK ASBESTOS FIBER ANALYSIS

June 30, 2004
_____
Effective through

_____
For the National Institute of Standards and Technology
NVLAP Lab Code: 200481-0

NVLAP-01C (06-01)

National Institute
of Standards and Technology

NVLAP®

National Voluntary
Laboratory Accreditation Program

ISO/IEC 17025:1999
ISO 9002:1994

Scope of Accreditation

BULK ASBESTOS FIBER ANALYSIS   NVLAP LAB CODE   200481-0

EMSL ANALYTICAL INC. MOBILE LABORATORY
107 Haddon Avenue
Westmont, NJ 08108-2799
Mr. Robert DeMaio
Phone: 856-858-4800   Fax: 856-858-1292
E-Mail: rdemaio@EMSL.com

NVLAP Code	*Designation*
18/A01	EPA-600/M4-82-020: Interim Method for the Determination of Asbestos in Bulk Insulation Samples

June 30, 2004

Effective through

For the National Institute of Standards and Technology

NVLAP-01S (06-01)

DEPARTMENT OF
PUBLIC HEALTH AND HUMAN SERVICES
ENVIRONMENTAL LABORATORY

JUDY MARTZ
GOVERNOR

GAIL GRAY, Ed.D.
DIRECTOR

STATE OF MONTANA

PHONE: (406) 444-2642
FAX: (406) 444-2617

1400 BROADWAY
PO BOX 4369
HELENA, MT 59604-4369

EMSL Analytical, Inc.
Attn: Ron Mahoney
107 West 4th Street
Libby   MT   59923

9/18/2003

MONTANA CERTIFICATE NUMBER: CERT0017

ATT: Ron Mahoney

I have received your application for certification for drinking water analysis in Montana, along with the needed information. Enclosed you will find a current certificate and a list of certified parameters. If you find any discrepancies on the parameter sheet please let me know and I will correct them. There may be analytes on the parameter list that are not regulated by the EPA for drinking water but may be regulated by the State of Montana. For out-of-state laboratories certified by Reciprocity, approved parameters will be the same as those granted by your home-state certifying authority.

The expiration date for your Certificate is:

CHEMISTRY: 03/18/2006

MICROBIOLOGY:

If you have any question concerning laboratory certification please contact me at (406 444-2642). Thank you for your time and prompt response in this matter.

Sincerely,

Judy Halm (Laboratory Certificiton Officer)

MONTANA DEPARTMENT OF
PUBLIC HEALTH AND HUMAN SERVICES
ENVIRONMENTAL LABORATORY

Recognizes that

EMSL Analytical, Inc.

of

Libby   MT

has completed the requirements for Montana certification and is licensed to analyze Montana's Public Drinking Water Supplies. See attached listing.

Montana Certification Number:	CERT0017

	Chemistry	Microbiology
EXPIRATION DATE:	09/18/2008	

Judy Halm
Judy Halm
Laboratory Certification Officer
DPHHS Environmental Laboratory

Date Issued: 9/18/2003

ENVIRONMENTAL LABORATORY

CERTIFIED DRINKING WATER PARAMETERS

EMSL Analytical, Inc.
107 West 4th Street
Libby, MT 59923
CERT0017
Certificate Expiration Date: 9/16/2006
Certified Drinking Water Parameter List

INORGANIC PARAMETERS

Analyte	Method 1	Method 2
Asbestos	EPA 100.2	

EMSL

ANALYTICAL, INC.

Outline of the
LABORATORY
QUALITY ASSURANCE
PROGRAM
For:

PHASE CONTRAST MICROSCOPY
TRANSMISSION ELECTRON MICROSCOPY
POLARIZED LIGHT MICROSCOPY

The Quality program at EMSL is built on a commitment to quality and continued improvement. This program is a primary part of our every day work; developed, utilized, and maintained by all the dedicated staff at EMSL.

CONFIDENTIAL

Introduction

This Program Outline provides a comprehensive overview of the Quality Assurance Program. It provides the reader with a summary of the Laboratory policies and procedures as they relate to the technical aspects of Corporate Quality objectives.

This program follows quality guidelines as documented by the American Industrial Hygiene Association (AIHA), the EPA's National Voluntary Laboratory Approval Program (NVLAP), National Environmental Accreditation Conference and other applicable state and federal regulatory agencies.

This QA program is designed to ensure that the highest level of quality professional services and technical excellence is provided to our clients. This is accomplished by the implementation of program policies including:

- Development of company standard quality control programs
- Standardization of reporting formats
- Review of regional laboratory QC performance
- Providing technical training for all staff levels
- Achieving traceability of data
- Performance of quality audits
- Participation in applicable Accreditation Programs
- Participation in applicable third party proficiency testing programs

The objectives of these program polices ensure the quality, accuracy and integrity of our analytical data.

The Quality Assurance objectives, policies and procedures are formally documented in the Quality Assurance Manual – EMSLQAASB101.6. An outline and summary of this manual is presented on the following pages.

Quality Assurance Program CONFIDENTIAL

The Objectives of the QA Program are to ensure the following:

To achieve these goals, this Manual directs implementation of the Quality Assurance Quality, accuracy and integrity of analytical results.

- Conformance with all analytical methodologies
- Conformance with Corporate mandated QA/QC requirements.
- Delivery of the highest quality of professional services and technical excellence to our clients.
- Fulfill the requirements of the American Industrial Hygiene Association, the National Voluntary Laboratory Approval Program, and/or the National Environmental Laboratory Accreditation Committee.

This Manual is to be kept accessible to all employees, and all employees are responsible for being familiar with, and adhering to its contents. Each employee is to sign the signature page acknowledging an understanding of the contents of this document. A copy of this signature page is submitted to the QA Department.

This Quality Assurance Program will be reviewed at least annually by the QA Manager. It will also be reviewed any time a problem arises that indicates a possible program flaw. In such an instance, the QA Manager will discuss the problem with Regional and Laboratory Management, Quality Control Supervisor and Analysts to ensure needed input from all levels within the Laboratory.

Implementation of the Quality Assurance Program

The program is designed to plan and institute Company policies and quality objectives throughout the branch laboratories. It is intended to provide support and issue policies including:

- Clear job descriptions delineating responsibilities of each employee involved at all steps of laboratory procedures, data analysis and report generation.
- Completion of Quality Control (QC) samples.
- Proper documentation of analytical data.
- Good laboratory technique that ensures a contamination-free environment.
- Use of appropriate analytical technology including review of current literature to capture recent applicable developments.
- Review of reports to Clients.
- Understanding and compliance with procedures which insure Client confidentiality

The program is managed and maintained by the Corporate Quality Assurance Manager as described in section 2.0 Organization and Responsibility.

It is our intention to ensure that all goals and objectives of our Quality Program are met and maintained. Quality policies and procedures are integrated into our daily work, and are constantly reviewed by Regional and Laboratory Management and by the Quality Assurance (QA) Manager

CONFIDENTIAL

Organization and Responsibility

EMSL Asbestos Laboratory

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                        ┌───────────┐
                        │ President │
                        └───────────┘
        ┌────────────────────┼────────────────────────┐
┌────────────────────┐ ┌──────────────┐ ┌──────────────────────────────┐
│Director of Sales & │ │General Manager│ │Director of Business Development│
│     Marketing      │ └──────────────┘ └──────────────────────────────┘
└────────────────────┘
┌────────────────────────┐              ┌──────────────────────────┐
│Quality Assurance Manager│              │ Quality Control Manager  │
└────────────────────────┘              └──────────────────────────┘
                        ┌──────────────────┐
                        │ Regional Manager │
                        └──────────────────┘
                        ┌──────────────────┐
                        │Laboratory Manager│
                        └──────────────────┘
            ┌──────────────────────────┐
            │Quality Control Supervisor│
            └──────────────────────────┘
┌──────────────────────────┐ ┌─────────┐ ┌──────────────────────┐
│Sample Receiving Coordinator│ │ Analyst │ │Data Entry Coordinator│
└──────────────────────────┘ └─────────┘ └──────────────────────┘
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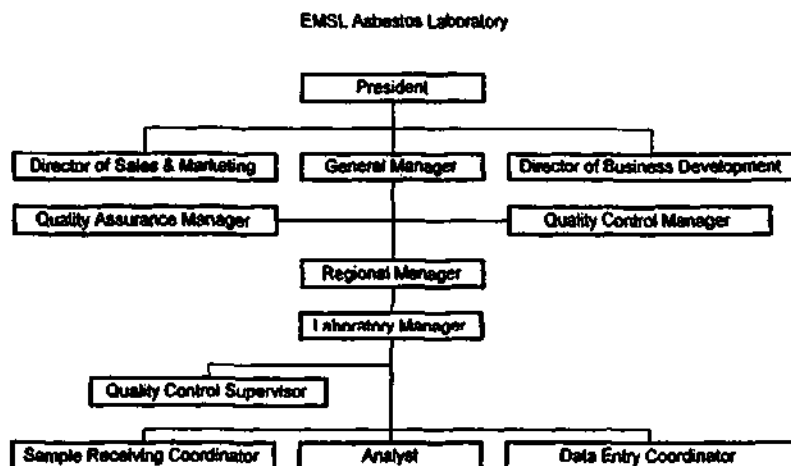
The Corporate Headquarters of EMSL Analytical operates out of the Westmont N.J. office location. The Corporate Headquarters over see the laboratory operations located there, as well as the Branch laboratory locations. Corporate Headquarters are responsible for the management of all company activities.

Branch laboratories (as well as the laboratory located in Westmont) perform the company's analytical services. They report to the Corporate Headquarters on Quality Control, productivity, staffing and market issues

Training
All analysts must complete the EMSL training program in order to perform such analysis independently. EMSL provides in-house training pertinent to all areas of analysis. This training is documented in the employee's record, and will be considered equivalent training for the specific method/authorization covered by the training.

Ethics
One of the objectives of the Quality Assurance Program is to insure the staff of EMSL is provided information in the aspects of ethics as they pertain to Corporate policy. The goals of this program are:

- For each staff member to understand the responsibility to provide true and accurate information
- The understanding of the consequences of unethical conduct
- Provide direction to employees
- Define right and wrong (as it is job related)
- The understanding of the impact of our actions

Standard Operating Procedures
Instructions or procedures for the activities affecting the quality of our analytical services, shall be developed by Management for their respective critical functions. This Quality Assurance Program shall be used as a guideline for their development, use and revision.

Technically specific Standard Operating Procedures are documented in the SOP manuals, located at each laboratory facility. These SOPs include step by step procedures for the preparation, analysis, and reporting of data.

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Acceptance of Work

Our services are generally offered as line item tests which reference documented methodologies. Laboratory services are typically requested by the client as "open order" request. Samples may be delivered to the laboratory at any given time, without a firm documented arrangement. Analytical services are often performed on verbal contract. In these situations, our general terms and conditions apply. The Management review procedures are established in the Quality Assurance program for these open orders, verbal contracts and for the cases where a written contract is utilized.

Sample Tracking

Chain of Custody

In order to ensure the integrity of any sample, records of its custody must be maintained throughout the sample collection in the field, acknowledgement of receipt, acceptance by the laboratory and analysis.

Since the client collects samples for analysis, the laboratory cannot be responsible for issuing a chain of custody at the time of sampling. However, the laboratory will advise all clients regarding sampling requirements (sampling materials, recommended sampling volumes, packaging, instructions for shipping, etc.) and chain-of-custody, and recommend that they use our form if they do not have their own.

Once the sample is accepted for analysis by the laboratory, the EMSL "Internal Chain of Custody" is used to document the handling of the samples throughout the analytical process.

Sample Acceptance Criteria

In addition to acknowledgement of the receipt of samples, samples must also be accepted for analysis. Prior to accepting samples, the person preparing the samples for analysis inspects them to determine if they conform to laboratory acceptance criteria. If they do not, or if this person has any question as to the validity of the sample, the Laboratory Manager or an analyst trained to analyze such samples will determine whether the questionable circumstance is sufficient to cause rejection. Rejections of samples are to be followed up by immediate notification to the client with an explanation.

Log In

Log in of samples is normally done by the Sample Receiving Coordinator, but may be done by any other employee familiar with the process. Information is entered for samples received into the Laboratory Information Management system (LIMS). LIMS is a computer laboratory management system which serves to track all samples from receipt through the analysis, reporting, and billing processes.

Archival and Disposal of Samples

Once the analysis is complete and the analysis worksheet is signed, the analyst stores the sample in the appropriate storage box, as indicated in the SOP. All storage boxes are to be stored in a safe manner for the period indicated for that category of waste, in accordance with regulatory requirements.

All bulk and air samples are held for a minimum of 2 months (60 days), unless a longer period is requested by the Client. All TEM grids are held for 3 years. Asbestos containing samples are disposed of by a licensed contractor, and a copy of the waste manifest is obtained and kept on file. If requested, samples will be returned to the Client.

Subcontracting

EMSL laboratories do not generally subcontract technical services. However, in the event such services are required, the Laboratory Manager will ensure all procedures are performed by laboratories that comply with the Quality systems as addressed in this document and the policies of the accreditation program(s) currently held by this laboratory. Laboratories must subcontract to outside laboratories that maintain accreditations appropriate for that analysis

Data Processing and Validation

EMSL utilizes an automated Laboratory Information Management System (LIMS) to record, document and assimilate pertinent field, laboratory, and administrative data. The validation of the software, including final report templates are performed by the Corporate MIS Department and the Quality Assurance Department.

Data validation is also a continuing process that takes place every time samples arrive at the laboratory and is carried through during log in, analysis and final reporting. This process is performed by the Laboratory Manager each time a final report goes through the procedures of review and signature.

Exported Data

Exported data is provided in a variety of formats, depending on the specific needs of our clients. Export formats for data deliverables are implemented and controlled by the corporate MIS staff, which has the flexibility; to implement new export formats as required. Electronically delivered data is not intended to replace hard copy results. Final, signed client reports are to be submitted in addition to delivery by email or diskette. In this way, exported data can be verified. Electronically transmitted results meet the requirements of the QA policies.

Record Retention Policies

The archived data must be retained as listed in the table below or as client contract requires:

AGENCY	RECORDS
NVLAP	3 YEARS
AIHA IH	3 YEARS
AIHA – ELLAP	10 YEARS
NYS ELAP	5 YEARS
water data	10 YEARS
California ELAP	10 YEARS
water data	
Texas Department of Health	30 YEARS
NELAP	5 YEARS

It is EMSL policy to store records for 5 years (if not otherwise contractually established). The following records shall be maintained:

- Copy of Chain of Custody Documents
- Original Analytical Data Recording Worksheets
- Quality Control Data
- All other records relating to the preparation of the client report

Quality of Materials

The high quality of materials used in this Laboratory shall be assured through specific purchasing and verification procedures and/or proper preparation techniques.

Selection of the appropriate grade of reagent(s) is designated in the reagent section of each analysis SOP and in addition may be specified by the Laboratory Manager in unusual circumstances. As a general practice, reagents will be of at least ACS reagent quality.

Reagents inclusive of SRM shall be purchased in accordance with the analytical needs of this Laboratory as determined by the Laboratory Manager. When received by the laboratory, these item's labels are dated and initialed with date received and expiration dates (if appropriate) as indicated /suggested by the manufacturer. Labels are also dated and initialed when opened and/or when reagent mixtures are prepared.

Verification will consist of confirming that the priority grade recorded on the reagent label conforms to the requirements of the SOP unless analysis difficulties indicate a possible problem or regulatory agency requirements specify otherwise. In the latter case, the appropriate analytical SOP will indicate the proper verification procedure.

Equipment/instrument maintenance

The quality and maintenance of equipment plays a critical role in providing quality analytical services. Maintenance schedules for equipment will be established by the Laboratory Manager. The Laboratory Manager shall also determine whether each microscope is maintained and repaired in-house or by an outside agency following EMSL administrative procedures. Servicing will also be performed when a need had been identified by calibration or other QC checks.

A maintenance file will be maintained for all equipment. In addition to a schedule of normal preventive maintenance, this file will contain a record of servicing.

Contamination Management

Proper observance of laboratory procedures is necessary to guarantee accuracy of results and the safety of Laboratory staff members.

Contamination both of samples and of the environment (including reagents used in analysis) must be avoided to provide the highest quality, legally defensible data to our clients. In order to achieve this goal, Laboratory staff must adhere to various preventative measures and use the testing procedures for contamination detection as established by the QA Manager.

If analysis of the blank samples indicate the possibility of contamination, the area and tools are cleaned and another sample prepared and analyzed. If analysis of the third sample shows contamination, a complete investigation is conducted to determine the contamination source. If contamination is detected in any situation, the source of contamination must be traced and the problem resolved to prevent reoccurrence. All procedures taken to resolve a contamination circumstance shall be documented properly and completely in the laboratory files.

Document preparation and control

In order to prepare and distribute documents in an organized fashion, procedures for initiation, preparation, review, approval and issuance of controlled copies will be followed. This program is a coordinated effort involving both technical review and custodial control. Analysts are to use only controlled, i.e., approved documents for all calibrations, analyses, final reports, and other activities performed in this laboratory.

Reporting results

The client report is ultimately, our "final product". The quality of our report reflects on our standard of quality. Final Client Reports are released only after data has been approved by the Laboratory Manager. This review includes Quality Control results, calibration measurements, etc. performed by the laboratory

Procedures for dealing with deficiencies

Any complaint by a client will be treated as a non-conformance, and treated with the same corrective action follow-up as a discrepancy seen in following internal Quality Control procedures.

If a client makes a complaint about a test result, the sample in question will be reanalyzed by a second Analyst. If the second result agrees with the original the Laboratory Manager shall advise the client in writing that a quality control check has confirmed the original analysis.

In all cases where a deficiency is discovered, the QA Manager will initiate a corrective action review to determine the root cause of the problem and action to take to prevent reoccurrence. A report will be issued to the Laboratory Manager, who is responsible for the corrective action implementation.

The corrective action will consist of a review of all steps leading up to the non-conformance. This will include review of QC data, sample tracking, data transcription, instrument calibration, training documentation, and discussion with personnel.

Following the review, the QA Manager will prepare a report detailing the cause of the error and corrective action to take to prevent re-occurrence. The QA Manager will also follow up on the corrective action to ensure its implementation

Analytical Performance Criteria

Performance criteria will be determined three ways:

1) Results from intra-lab and round robin testing will be plotted to see if they fall within warning and action limits.

2) The administering agencies for proficiency testing will determine performance criteria.

3) Achievement of internal on-site Quality audits by the Regional or QA Manager. These audits will verify compliance with all QA and QC policies as documented in this manual. The Quality Audit process is detailed below.

Quality Control is performed continuously throughout the course of laboratory sample analysis regardless of laboratory productivity and is made part of the normal course of laboratory sample analysis.

Frequency and volume of QC analysis is based on regulatory requirements and Good Laboratory Practice. These requirements are listed for each analysis type in Appendix A of this manual. These methods will be used according to the scope of the laboratories accreditation status and quality control requirements for each type of analysis. Performance criteria will be maintained for both individual analysts and for the entire laboratory. The standards for acceptance criteria are documented in the EMSL Quality Control Standard Operating Procedure Manual, *EMSLQCPGRMSOP.200.x.*

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Quality Audits

Quality Audits will be performed for each laboratory location on an annual basis or more frequently as deemed necessary by Corporate, Regional or Laboratory Management. Audit procedures and policies are issued by the Quality Assurance Department and include:

- Review of compliance with the Quality system
- Compliance with Quality Control analysis
- Identification of any problem areas and suggestions for resolution

The Quality Assurance Department develops the guidelines and overall manner by which a Quality Audit is performed. These policies are detailed in the Standard Operating Procedure for Quality Audits, *QAASBAUDITSOP.200.0.*

Management Reviews

Management reviews are designed to provide the Corporate Management of EMSL with an overview of the performance of the QA activities of the laboratory operations. It addresses the Quality topics documented in ISO 17025 for each laboratory location and includes:

- Quality control activities
- Outcome of recent internal audits
- Asessments by outside agencies
- Corrective actions
- Major changes in personnel, work load, service areas
- Client dealings

Proficiency Testing Programs

Laboratories participating in proficiency testing programs will insure the analysis is performed using the same analytical methodology and staff as under normal, client sample conditions. At no time is there inter-laboratory exchange of samples.

Records of proficiency testing analysis are to be completed and maintained in a separate laboratory PT file. This data is also maintained for each participating analyst in his or her personal training file.

Demonstration of Traceablilty

The Quality Assurance program is designed to provide a method which achieves traceability of data to national standards. This is accomplished by setting requirements which include:

- Use of Standard Reference Materials as certified and traceable to the National Institute of Standards and Technology. SRMs are used for QC analysis and training for achieving measurements of analysts and overall laboratory accuracy.
- Calibration of instrumentation against NIST traceable standards
- Laboratory participation in independent (non EMSL) proficiency testing programs
- Analysis of consensus standards

Client Communications

Clear, continuous and open communication between the laboratory and the client is one of the keys to maintaining a successful, quality operation. Communication should be established prior to the start of any work. Information should be clearly understood between the Laboratory Management and the client. EMSL provides Quality Assurance information and technical support to the client to assure continued quality service. The support and information provided in relation to the work performed includes:

- Field sampling guides
- Availability of pertinent QC records
- Access to the Quality Assurance Department for technical assistance
- Security of data (confidentiality)
- Reasonable access to the relevant areas of the laboratory for the witnessing of analysis

Confidentiality

It is understood that confidentiality and proprietary rights must be respected throughout the performance of services for any client or for those that may include national security concerns. Information will not be given to those for whom it is not intended and the proprietary rights of our client will be protected.

Notice of Performance

The Laboratory Manager should provide the client with information as it relates to the performance of the analysis and turnaround time. The laboratory must notify the client if:

- Analysis cannot be performed on time
- Integrity of the sample has been jeopardized (either by the laboratory or the client)
- A discrepancy in the analysis has been found during QC analysis.

Record of conversation is generally documented on the chain of custody. The person making the entry dates and initials the record.

Analytical Quality Control Programs

The Quality Control program as established and managed by the QA Manager ensures that this Laboratory produces quality data. This process ensures, at a minimum, that our data is legally defensible and that all personnel perform their responsibilities properly.

The Laboratory Manager will determine how QC testing is implemented operationally (e.g., after the analyses of every ten samples or at the end of each day, etc.) QC analysis is performed on a minimum of 10 % of sample volume. QC testing occurs on a regular basis and is not scheduled around the amount of workload.

In addition , the QA Manager will inspect the results of all QC testing on a regular basis and provide the necessary support and directives to the Laboratory Manager to ensure the QC program is properly executed.

Quality Control - General

Our laboratories internal QC program includes at a minimum, 10% quality control on all samples received for analysis. These are summarized below in each analytical section and include:

- Analysis of standard reference materials
- Intra analyst QC
- Inter analyst QC
- Analysis of blank samples
- Participation in inter laboratory programs
- Participation in proficiency testing programs

This QC data is graphed on control charts designed specifically for each analysis type. The description of these control charts are detailed in the Standard Operating Procedure EMSLQCPROGSOP.200.

The Laboratory Manager (or Managers designee, i.e. QC Supervisor) of each laboratory is responsible for implementing the day-to-day QC testing and ensuring the correct types of testing occurs at the appropriate frequencies. The Laboratory Manager is also responsible for ensuring complete records of QC testing are maintained.

Training - General

New analysts with no prior formal training must complete the EMSL training program in asbestos analysis in order to perform such analysis independently. The Lab Manager will draw on the candidate's previous training, if any. The candidate will receive sufficient in-house training to demonstrate proficiency and understanding in all related topics to the Lab Manager's satisfaction.

Practical Factors: When the candidate has received sufficient training to analyze samples, he/she will work in the laboratory along side an experienced analyst. The candidate will not sign any reports. All samples will be checked by an experienced senior analyst, who will officially report the results for review and signature, by the Laboratory Manager.

Proficiency Analysis: The candidate will be deemed proficient when quantitation within l laboratory norms, as established by the QC schedule are met.

Additionally, the trainee must perform analysis on past proficiency samples and succeed in generating data within the acceptable range as established by the agency(ies) statistical analysis. (see training SOP for additional detail)

Records are kept of the candidate's progress by the analyst-training log. When all areas are signed off, the candidate may perform independent analysis.

Phase Contrast Microscopy (PCM)
Method following: NIOSH 7400
The Quality Control program for phase contrast microscopy includes intra-analyst sample testing, participation in inter-lab programs, and statistical evaluations and calibrations.

Calibration

Calibration procedures must be followed prior to the analysis of air samples to insure that results of analysis reflect true and accurate data. The following summarizes the type and frequency of calibration for the analysis of fibers in air by PCM. Details of these procedures are found in the PCM SOP, *EMSL.XXPCMSOP.200.x.*

1) Microscope calibration
  Phase Ring Alignment
  Contamination control
  HSE/NPL Test Slide
  Measurement of Walton Beckette Graticule

2) Analysts calibration
  Standard reference slide (past Proficiency test slide)

3) Operational Calibration
  Air monitoring
  · Hood calibration

Details on the calibration procedures for PCM can be found in the Standard Operating Procedures Manual.

Quality Control Analysis

Each analysis performed in this Laboratory has its own *Quality Control requirements that have been* determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

  Intra-Analyst
  Reference
  Proficiency Testing
  Round Robin Testing
  Laboratory Blank Analysis

The Laboratory Manager (or Managers designee, i.e. QC Supervisor) of each laboratory is responsible for implementing the day-to-day QC testing and ensuring the correct types of testing occurs at the appropriate frequencies. The Laboratory Manager is also responsible for ensuring complete records of QC testing are maintained.

Transmission Electron Microscopy (TEM)
Method following: AHERA - 40 CFR, Part 763, Subpart E, EPA Level I, II, III - EPA Contract # 68-02-3266, ASTM D 5755-95

The QA/QC program for the analysis of asbestos via TEM insures compliance with standard regulatory guidelines and follows Good Laboratory Practice (GLP). The program includes:

- Achievement of Verified Status
- Classification of Structures
- Calibrated Measurements at .5 micron
- Calibrations
  - alignments
  - magnification
  - camera constant
  - plasma asher
  - detector resolution
  - grid opening measurements
  - analytical balance
  - muffle furnace
- Fiber Id and Sizing
- SAED Indexing
- Ambient air monitoring

Quality Control Analysis
Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

> Intra-Analyst
> Intra-Analyst repreparation
> Inter-Analyst
> Reference Standards
> Verified Analysis
> Proficiency Testing
> Round Robin Testing
> Laboratory Blank Analysis

Polarized Light Microscopy
Method following: EPA-600/R-93/116, EPA-600/M4-82-020
Quality control procedures in the PLM laboratory follow guidelines as documented by the NVLAP accreditation program.

Calibration procedures must be followed prior to the analysis of samples to insure that results of analysis reflect true and accurate data. The following summarizes the type and frequency of calibration for the analysis of asbestos in bulk materials by PLM. Details on the performance of these functions are found in the PLM SOP.

1) Microscope calibration
   > Center Stage or objective, & condenser
   > Align polars
   > Crosshair alignment fixed in polarizer's privileged direction

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2) Analysts calibration
> Standard reference sample
> Contamination check with fiberglass sample
> Check Standard Amosite mount for proper dispersion colors,
> refractive index

3) Operational
> Calibrate Analytical Balance
> > Air monitoring
> > Refractive mounting oil calibration
> > Calibrate muffle furnace temperature
> > Hood calibration

Details on the calibration procedures for PLM can be found in the Standard Operating Procedures Manual.

Quality Control Analysis
Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

> Intra-Lab Testing
> Inter Analyst
> Intra-Analyst
> Reference Standards
> Proficiency Testing
> Round Robin Testing
> Laboratory Blank Analysis

Appendix 7

- Badlands Environmental Consultants, Inc., Quality Manual, 1/5/00

BADLANDS ENVIRONMENTAL CONSULTANTS, INC.

Quality Manual
01/05/00

Initiated by: _____

Mark Emter, Laboratory Director
Laboratory Supervisor
PCM Analyst
Quality Assurance/Quality Control Coordinator

Approved by: _____

James D. McGurren
Chief Executive Officer

Badlands Environmental Consultants, Inc.
1006 East Central Ave.
Bismarck, ND 585801
(701) 223-7335

TABLE OF CONTENTS

INTRODUCTION

Badlands Environmental Consultants, Inc. (Badlands) is a full-service, independent environmental health and safety consulting firm founded in Bismarck, North Dakota in July, 1999. Badlands provides scientific and technical expertise in industrial hygiene and environmental management to a wide-ranging client base including business, industry, and government. The firm was founded to provide clients with solutions and guidance in managing and monitoring their environmental and occupational health and safety needs. Our team of scientific professionals can provide unique and innovative solutions to a variety of environmental health and safety challenges.

Badlands's Quality Assurance (QA) Program was developed and implemented to provide analytical data of known and supportable quality to ensure a high professional standard is maintained with respect to analytical data generated. The goals of this QA Program is accomplished by:

- Providing a consistent framework for generation of analytical data.

- Establishing standard procedures that permit interlaboratory comparison of data.

- Establishing procedures for demonstrating that analytical protocols are followed and analytical systems are in control.

The objectives of QA Program are to:

- Verify the quality of Phase Contrast Microscopy (PCM) systems for precision and accuracy sufficient for the needs of each type of project.

- Assist in early recognition of deficiencies that may effect the quality of data.

- Enable the analysts/laboratories to identify and implement actions that are necessary to ensure the validity of data.

  - Ensure sufficient documentation to verify the quality of data submitted to the client.

SECTION 1

DOCUMENT CONTROL AND REVISIONS

Section 1: Document Control and Revisions

Introduction

A laboratory quality assurance program is the documentation that demonstrates acceptable laboratory practices are being followed. The system used for this quality manual is described below.

Indexing Format

This quality manual uses a standardized indexing format that provides for convenient replacement of pages that may be changed.

The indexing format includes, at the top of each page, the following information:

- Section number
- Page number
- Revision number
- Date of revision

Revisions

For each subsection, the text begins on a new page. This format groups the pages together to allow convenient revision of the subsection. Each time a new page is added or an old page expanded within a subsection, the number of the preceding or original page is included on the new page, and a letter is added to it. For example, if in Section 2, page 3 of 6 was revised and expanded to include an extra paragraph, any overflow would appear on a page designated 2.3a. The original page 4 would then be removed from the handbook and replaced by revised page 3 and 3a. This allows expansion within a section without replacing the section or renumbering all of the pages. The revision number will increase and a new typed page date will be entered.

Distribution Record

A distribution record will be maintained so that additions and revisions to existing handbook sections can be distributed to handbook users.

Responsibility for Changes

Changes may be made by the issuance of an entirely new document, replacement pages, an errata sheet, or by pen and ink posting on the original document. Upon any of these changes, all revised pages, sections, etc. will be removed and destroyed. Changes may also be made by pen and ink correction as indicated by the initials of the individual making a change.

SECTION 2

SAMPLE CUSTODY AND RECEIVING

Sample Custody and Receiving

An individual is designated sample custodian at the laboratory.   This person is responsible for the inspection, unpacking, laboratory number assignment and also scheduling of analysis.   Other personnel have been trained and are supervised in all receiving procedures as well.

Upon receipt of any samples to the laboratory the following receiving procedures are strictly adhered to:

   a.   The condition of the shipping package and any written data are noted upon receipt.

   b.   All bills of lading or shipping slips are retained to document the shippers and delivery times.

   c.   If any substantial damage to the package is observed or chain of custody seal (if any) is broken no action to unpack the shipment should be taken until a responsible lab manager is notified of the situation.

   d.   All shipments are unwrapped in a clean spacious facility.   The contents are recorded, including a brief description of each item and all identifying numbers or corresponding data.

   e.   After data and sample correlation are completed, a laboratory client number and job number are assigned to each sample and it is scheduled for analysis.

All Chain-of-Custody samples submitted to the laboratory are carefully monitored and their integrity is ensured and documented by the Quality Control Coordinator.

Sample Acceptance Criteria

   a.   All samples are isolated from one another in airtight containers.

   b.   All containers are intact and no physical evidence of debris material is observed.

   c.   Sample numbers correlate to number on applicable paperwork.

   d.   All pertinent paperwork, including any chain-of-custody forms is submitted with samples.

Unless all the above criteria are met, no samples of suspect asbestos content will be accepted into the laboratory for analysis.   If upon notification of deviations from

acceptable criteria, client resolves problems, samples will be accepted for analysis under normal conditions.

Sample Group Handling Protocol

When samples are received for analysis, the sample custodian will identify the type of samples, log them in and turn in a copy of the paperwork to corporate administration.

The samples are then prepared by the sample custodian or analyst for the requested analysis. The samples are then analyzed and archived. The analyst or lab director inputs results into the computer. If the analyst or lab director finds mistakes, the report is returned to the analyst for corrections. After corrections are made, the report is reviewed a second time before being sent for signature. The analyst signs the typed sheets and gives the paperwork to the QA monitor for a final review. If mistakes are found by either the analyst or QA monitor, the paperwork returns to the analyst who makes corrections and returns the paperwork to the individual for his or her signature, and the cycle is repeated.

After the analyst signs the paperwork, the paperwork is routed to the proper department. If the sample group has an associated project, the sample custodian gives the paperwork to the manager of that project who files the bench sheets with the other project paperwork. The office manager checks reports with cover pages. After the cover pages are checked, the report is mailed, filed and invoiced through the California office.

Section 2.3: Subcontracting of Calibration and/or Testing

Badlands will send samples <u>only</u> to NVLAP and AIHA accredited laboratories when the laboratory cannot fulfill its obligation to the client. Badlands will include in the report a statement indicating that a subcontracted laboratory completed testing or calibration.

Sample criteria and chain of custody will be maintained through Badlands policy. All subcontracted reports will contain the following statement:

"This report contains data which were produced by a laboratory subcontracted to Badlands Environmental Consultants, Inc. (NVLAP Lab Code) for the calibration or test methods performed."

"This report contains data which were produced by a laboratory subcontracted to Badlands Environmental Consultants, Inc. (AIHA Lab Code) for the calibration or test methods performed."

SECTION 3

ANALYSIS

Definition and Mineralogy of Asbestos

Asbestos is a generic term for a group of naturally occurring fibrous mineral silicates. As minerals, they crystallize in narrow veins as parallel bundles of extremely fine fibers. As the result of physical degradation, these compact bundles break down into finer bundles and individual fibers. Perfect lengthwise cleavage is the main characteristic of asbestos and high length-to-width ratio of individual fibers is a necessary criterion for a mineral to be called asbestos. Some fibrous forms of a given mineral have special names while others are simply called fibrous or non-fibrous forms of a particular mineral.

Asbestos terminology:

Asbestos Mineral	Non-Asbestos Mineral
Chrysotile	Lizardite and Antigorite
Fibrous Tremolite	Non-fibrous Tremolite
Fibrous Actinolite	Non-fibrous Actinolite
Fibrous Anthophyllite	Non-fibrous Anthophyllite
Amosite	Cummingtonite and Grunerite
Crocidolite	Riebecktie

The asbestos minerals occur in two mineral classes: serpentines and amphiboles.

Serpentines

This group of minerals is characterized by a layer lattice structure in which SiO_4 tetrahedral are arranged in sheets separated by sheets corresponding in composition and structure to the mineral brucite (Mg (OH^2). In chrysotile, the mismBadlandsh between the silicate and brucite sheets results in the sheet structures rolling up into scrolls referred to as "unit fibrils" of chrysotile. Chrysotile fibers are actually bundles of several thousand of these unit fibrils.

Amphiboles

The other asbestos types belong to the amphibole class of minerals, also known as double chain silicates. In the amphiboles the SiO_4 tetrahedra are linked in a double width chain which could have infinite length. It is development along the length of this chain that results in the fibrous morphology of the asbestiform amphiboles.

There are two main forms of asbestos that are amphiboles, amosite and crocidolite, along with three less frequently encountered types: tremolite, actinolite and anthophyllite. Both amosite and crocidolite were used most extensively as building materials in high temperature applications. Whereas tremolite, actinolite and

anthophyllite were used considerably less frequently and usually appear as components of other filler materials, notably some industrial talc.

SECTION 4

QUALITY ASSURANCE PROCEDURES

Badlands participates in the American Industrial Hygiene Association ProficiencAnalytical Testing (PAT) Program for air sample analysis via Phase Contrast Microscopy. On a quarterly basis four referenced samples are received, analyzed, and known results are compared to analyst's reported values. All results are maintained in each analyst's personnel file.

Corrective Action of Detected Test Deficiencies

When a defect is detected for an individual analyst, the laboratory supervisor or quality control coordinator will discuss the error with the analyst and review the sample with him. The sample or ones of similar composition will be submitted to the analyst for blind reanalysis until proficiency is determined. Any consistent errors made by an analyst will be noted in their personnel file along with corrective action.

Internal System Audits

The Quality Control/Quality Assurance officer conducts an annual audit of the laboratory. Items covered during the audit include 1) sample maintenance 2) calibration 3) preventative maintenance 4) analytical methods 5) data verification 6) record management.

The audit shall consist of a general and specific method/procedure audit. A General Audit shall be an overview of the laboratory as a whole. This audit will cover from sample receipt to report generation. A specific method/procedure shall be a detailed, in-depth review of an actual method of procedure.

After the audit has been conducted, the Quality Control/Quality Assurance officer shall complete an audit form. Any problems, observations and findings found will be discussed with the Laboratory Director. The results will be kept on file along with any corrective actions taken.

SECTION 5

CONTAMINATION CONTROL

Section 05: Contamination Control

Materials Returned to the Client

Upon the request of a client to have their samples returned, the following steps will be followed and adhered to by authorized BADLANDS laboratory personnel:

A. Complete a new Chain of Custody, retain laboratory copy and attach with original paperwork.

B. Return samples in original container and double bag.

C. Label container with appropriate BADLANDS lab numbers for tracking purposes.

D. Retain portion of sample for proof of results reported.

SECTION 6

RECORD KEEPING SYSTEM

Section 6:   Record Keeping System

Air Analysis Reports

All results of samples analyzed for asbestos content are kept on file in separate files according to the client name. All originals of results are kept in separate files in the master file room. . On a monthly basis, a second analyst will review and verify 10% of the data produced by the laboratory. The second analyst will initial the log in form and bring to the attention of the QA Coordinator any non-compliance.

Blank Sample Analysis Report Forms

The analysis of blank samples for contamination control are recorded on separate forms that are retained along with the other quality control forms in a laboratory binder. These results are summarized at the end of every month to evaluate the ongoing condition of the laboratory and/or the reagents used in analysis.

Testing Complaints from Clients

When a client questions a test result, the sample in question will be pulled and reanalyzed. If an error has occurred, any samples that are similar from the same batch will also be rechecked. Information including client name and number, sample description, results of first and second analysis and analysts' names will be recorded and filed. The client will receive an amended report containing the necessary corrections.

If consistent errors are noted for a particular analyst or a particular type of sample over a period of time, corrective action will be initiated.

Calibration Records

Calibration of the PCM microscopes is performed daily and the records are kept on a clipboard beside each microscope. At the end of each month the record sheets are placed in a laboratory notebook along with maintenance and repair information, if applicable. A file is kept for the hoods and muffle furnaces to document calibration, maintenance and repair.

Quality Manual Internal Audit

On a quarterly basis the quality manual will go through an internal audit which will be conducted by the Quality Assurance Monitor. All laboratory procedures and quality control documentation will be reviewed and noted on an BADLANDS laboratory checklist. The Quality Assurance Monitor will forward all pertinent findings to the Laboratory Director upon compilation of the audit.

Review of Quality Assurance System

The quality assurance manual will be reviewed on an annual basis and revisions for each year will be noted. Conditions that may require an immediate revision in quality assurance procedures during the course of the year will be listed and dated when they are recorded in the manual.

Summary

All records of laboratory analysis and the associated paperwork are retained for three years in standard file cabinets with back up on computer disc and hard drive. Only original paperwork will be in the hard copy form. This action should improve both client confidentiality and protection of lost or damaged paperwork.

SECTION 7

LABORATORY POSITIONS

Section 7:   Laboratory Positions

Position: <u>**Laboratory Directory**</u>
         Mark E. Emter

Responsible to the Chief Executive Officer of Badlands, Bismarck, North Dakota. Has overall responsibility for laboratory management and services. Duties include but are not limited to supervision of Badlands laboratory, field inspection and field technician personnel. Responsible for the training of the above-mentioned personnel, quality control and quality assurance programs, analytical procedures, maintenance of equipment and analyses.

Position: <u>**Laboratory Supervisor**</u>
         Mark E. Emter

Maintains operation of laboratory on day-to-day basis and supervision of all personnel inclusive of laboratory facility. Responsible for maintenance of quality assurance parameters and general flow of laboratory paperwork. Maintains employee files and is involved in personnel interviewing and acquisition. Responsible for ordering of supplies, analysis equipment and all field equipment utilized in obtaining air and bulk material samples. Maintains accreditations and certifications, handles correspondence and communications with accrediting/certifying organizations. Establishes and maintains interlaboratory sample exchange programs. Documents QA/QC operations manual and updates manual annually.

Position: <u>**QA/QC Monitor**</u>
         Mark E. Emter

Assigns analysts to analyze proficiency testing samples and establishes system for effecting blind analysis of such samples. Monitors results, resolves related problems or deficiencies. Maintains record keeping system. Oversees training analysts: 1) plans for external training courses, 2) develops follow-up internal training procedures, 3) supervises in-house training, and 4) documents training progress. Identifies critical factors to be charted, develops and supervises process for assignment (or blind analysis) of duplicated analyses. Determines frequency of analysis of knowns, e.g., PATs, AAR, etc. Monitors analyst compliance with QC program. Monitors currency of records, e.g., PM tables, control charts. Documents and resolves deficiencies. Handles analysts' questions on QC related problems. Resolves out-of-control point problems. Provides QA/QC program orientation to new analysts. Reports periodically to laboratory supervisor: 1) verbally reports significant problems immediately, 2) provides monthly formal summary reports on progress, and 3) provides data for analyst and laboratory QC related performance measurements, e.g., number of lost samples, number of internal and external out-of-control points.

Position: Laboratory Analyst
        Mark Emter
        James D. McGurren
        Patrick McGurren
        Daniel McGurren
        John V. Brock
        Michael J. Mithun

Responsible for analysis of air samples by Phase Contrast Microscopy. Laboratory duties include the following: 1) general maintenance and calibration of analytical equipment, 2) maintenance of laboratory cleanliness and contamination control, 3) inventory and organization of laboratory supplies including analytical solvents and reagents, 4) sample log-in and chain-of-custody maintenance throughout sample analysis procedures, and 5) storage and archiving of samples and sample analysis data. Also responsible for analytical data review and editing of erroneous information in analytical reports. Answers directly to both laboratory supervisor and QA/QC monitor.

Position: Laboratory Technician

The following is a description of tasks performed by the Badlands staff members responsible for air sample preparation.

Position: Sample Custodian
        Mark E. Emter

The following is a description of the tasks performed by Badlands staff during sample check in.

Air samples are received at the laboratory via mail, walk-in delivery or any of the delivery services available to our clients. Laboratory personnel under a protective hood open packaging. Samples that are accepted by the laboratory for analysis must be accompanied by a completed chain-of-custody which includes the signature of the person relinquishing the samples, the turnaround time for the samples and a description of the samples. The laboratory analyst/ assistant who opens the sample packaging then signs the chain-of-custody with the date and time of receipt after the number and description of the samples have been verified. The chain-of-custody is then routed to the laboratory staff so that the sample batch may be given a project code number (PCN). If the samples are intercompany, the sample batch is labeled with the project code number and the chain-of-custody remains with the samples.

SECTION 8

TRAINING REQUIREMENTS

Section 8: Training Requirements

Laboratory Technicians

An experienced analyst (i.e., laboratory supervisor or quality control coordinator) will train all lab technicians involved in sample preparation of samples. Hands-on training follows. Points emphasized are:

a. Making a good representative slide of each sample.
b. Careful organization of samples, slides and paperwork.
c. Maintaining a contamination-free work area.

The health hazards of asbestos are explained and the importances of maintaining safety precautions are emphasized.

All personnel involved in asbestos-related work are given a physical examination in accordance with OSHA regulations for asbestos workers. These examinations are given prior to any performance of work and on a yearly basis thereafter.

SECTION 9

LABORATORY REFERENCE DOCUMENTS

Section 9: Laboratory Reference Documents

Laboratory Reference Documents

1. 40 CFR Part 763, Volume 52, No. 210, "Asbestos Containing Materials in Schools, Final Rule and Notice."

2. "NIOSH 7400 Method", issued 2/15/84, Revision 3, 5/15/89.

SECTION 10

LABORATORY FORMS USED FOR QUALITY CONTROL

SECTION 11

PUBLICATION HISTORY

Revisions

First Printing
    Quality Manual – January 5, 2000

First Revision
    Quality Manual

Second Revision
    Quality Manual

Third Revision
    Quality Manual

ANNUAL REVIEW

Date	Reviewed By	Signature	Title

DIRECTOR REVIEW

Date	Reviewed By	Signature	Title

SECTION 12

STANDARD OPERATING PROCEDURES

Section 12: List of Standard Operating Procedures

Standard Operating Procedure	Revision	Date
SOP 01: Laboratory Safety	1	4-98
SOP 02: Sample Custody	4	4-98
SOP 03: Sample Log-In	1	4-98
SC-3, SOP for Sample Log-Out	4	4-98
SP-2, SOP for Air Sample Prep	4	4-98
SA-2, SOP for Air Sample Analysis	4	4-98
RG-2, SOP for Air Sample Analysis and Reports	3	4-98
QC-2, SOP for QC of Air Samples	4	4-98
QC-3, SOP for Corrective Actions	4	4-98
QC-4, SOP for Interlaboratory Air Samples	3	4-98
QC-5, SOP for Monthly/Quarterly Reports	2	4-98
QA-1, SOP for Preparation Hoods	2	4-98
QA-4, SOP for Wipe Sampling	4	4-98

Standard Operating Procedure	Revision	Date
QA-5, SOP for Laboratory Air Monitoring	3	4-98
QA-8, SOP for PCM Microscope Calibration	3	4-98
QA-9, SOP for Ambient Air Temperature	2	4-98
QA-10, SOP for Annual Audits	1	4-98
QA-11, SOP for Adopting, Reviewing, and Raising Analytical Methods and SOP's	1	4-98
TR-1, SOP for Training a New Analyst	1	7/98

Key to SOPs
TR = Training
LS = Laboratory Safety
SC = Sample Custody
SP = Sample Preparation
SA = Sample Analysis
RG = Report Generation
QC = Quality Control
QA = Quality Assurance

SOP 01: STANDARD OPERATING PROCEDURE FOR LABORATORY SAFETY

Unsafe acts and/or unsafe conditions cause accidents. All personnel will assist laboratory management in identifying and correcting unsafe conditions. Additionally, personnel must conduct all job duties in such a manner that they do not endanger themselves or others.

General Safety Practices

1. Learn the correct way to perform a procedure. Read the instructions provided and ask questions.

2. Work at a speed consistent with safety.

3. Obey warning signs and tags.

4. Do not engage in horseplay.

5. Report unsafe conditions to your supervisor.

6. Know the location of the nearest fire extinguisher and emergency exit.

7. Use protective equipment (lab coat, gloves, face shield, etc.)

8. Always wear safety glasses in laboratory areas.

9. Handle volatile, toxic, and/or noxious materials in an appropriate fume hood.

Housekeeping

Cleanliness, order, safety and efficiency are inseparable. A cluttered workplace does not indicate the amount of productive work occurring.

1. Keep aisles clear at all times.

2. Maintain clear passageways to emergency equipment, exits, showers, eyewash fountains, and electrical control panels.

3. Store equipment and supply materials in appropriate locations that do not endanger yourself of others.

4. Do not drape electrical cords or hoses across aisles.

5. Clean floors immediately when liquids are spilled.

Electrical Safety

Show proper respect for electrical systems. Most electrocutions occur with 120V AC. The higher the voltage the more danger involved but it is the current and current path which determine the severity of tissue damage. The three hazards from electricity are:

A) Shock
B) Burn
C) Effect of pulling or jumping back

1. Check the insulation on the electrical cord when working with portable electrical devices.

2. Know where and how to cut the electrical power to the area you are working in.

3. Do not remove the grounding prong from male plugs.

4. Do not use electrical equipment if your hands are wet or if you are standing on a wet surface.

Chemical Safety

The hazards associated with chemicals are corrosive burning of the skin, defatting the skin, and dermatitis; internal injury and systemic poisoning from inhalation, ingestion, or absorption through the skin; and damage from the physical effects of heat and shock caused by explosion and burning.

1. Unless you intend to ignite them, keep flammable materials away from heat and strong oxidizers. The vapors of many flammable solvents are heavier than air and will flow and accumulate in low spots.

2. Use adequate ventilation/fume hood.

3. Avoid breathing vapors.

4. Avoid contact with skin and eyes.

5. Wear appropriate personal protective equipment

6. Identify chemicals and solvents and label their containers.

7. Use proper containers; do not store chemicals in coffee cups or drink coffee from beakers.

8. Report chemical spills promptly to your supervisor.

9. Know the location of and how to use the nearest eyewash fountain and safety shower.

Asbestos Safety

All samples submitted to BADLANDS for asbestos analysis may contain asbestos. Samples and prepared slides must be handled carefully and cautiously at all times.

1. Open sample containers in a Class I Biohazard hood.

2. Exacto knives are sharp - use with care.

3. Asbestos is carcinogenic! Do not inhale or swallow any fibers.

4. Wash hands after mounting asbestos filter material.

Office Safety

An office is a relatively safe place to work but the accidents that do happen can be as painful as those that occur elsewhere.

1. Keep floors clear of rubber bands, pens, pencils, paper clips, and paper. Any of these can cause slips and falls.

2. Do not climb chairs or reach beyond your physical limits.

3. Open one file drawer at a time.

4. Close desk drawers and file drawers as soon as you no longer need them open.

5. Do not drape extension cords or telephone cords over partitions or run them across floors.

Accidents

In spite of our best efforts and intentions to eliminate accidents, sometimes disaster strikes. When it does:

1. Report accidents to your supervisor immediately. Also report any property damage.

2. All personal injuries, regardless of how minor, must be reported within three (3) days. Failure to do so could result in a loss of State Compensation Insurance Fund benefits.

Fire and Other Emergencies

You should know the location of the nearest fire extinguisher. Calmly but quickly use the carbon dioxide type extinguisher on the fire. This type of fire extinguisher is suitable for type A, B, or C fires. These are:

A - Wood, paper, textiles
B - Oils, greases, solvents
C - Live electrical equipment

If possible to do so without endangering yourself, turn off any live equipment at the main switch immediately.

Notify your supervisor and co-workers as quickly as possible. If required, have someone notify the fire department; the fire department phone number is on the back of all telephone receivers.

SOP 02:   STANDARD OPERATING PROCEDURE FOR SAMPLE CUSTODY

1. Forms

   a)   All chain-of-custody forms submitted with samples are signed and dated by the laboratory analyst/assistant who opens the sample packaging. The chain-of-custody is signed if no discrepancies exist between client data sheets and the actual number and description of samples; or when discrepancies are rectified. The chain-of-custody is then routed to the administrative staff so that the sample batch may be given a sample code number (SCN). If the samples are intercompany, the sample batch is labeled with the project code number and the chain-of-custody remains with the samples.

   b)   Chain-of-custody forms remain with the samples until they have been analyzed. Copies of the signed custody forms are sent to the client with the written reports, and the original is filed with our copy of the written report.

   c)   If a client requests samples to be returned, the original chain of custody is pulled from the files, signed, dated, copied, and then sent back to the client with the samples.

2.   Samples

   a)   Samples are archived by BADLANDS for a twelve-month period by the following procedures:

      i.   Samples are boxed by project code numbers for temporary storage immediately following analysis.

      ii. Sample custodian transfers samples to storage for permanent storage and files them by client name and project code number.

   b)   Unless prior arrangements have been made with the client, samples are turned over to a local abatement firm for safe disposal after the twelve-month holding period. At the time of waste collection, a waste shipment record will be completed to verify correct action was taken prior to disposal.

   c)   When samples are returned or sent for disposal, the information is recorded on the project code number for future reference.

SOP 03: STANDARD OPERATING PROCEDURE FOR LOG-IN OF ASBESTOS SAMPLES

1.   Samples and Data Sheets

    a)   Carefully remove staples from packaging making certain no bags, vials, etc., are opened (this process should be done under a high efficiency hood). Samples should be rejected if the container was opened during transit. Check the accept/reject column for each sample.

    b)   Make sure sample numbers on client's data sheets correspond with sample numbers on sample containers. If samples are missing or damaged, call the client immediately and record deficiencies on data sheet.

    c)   If the sample data sheet is also the client's chain-of-custody, it should be signed and dated by the laboratory analyst/assistant who opened the package.

2.)   Log-in

    a.)   BADLANDS job numbers are created from the existing project number assignment system. The following information is typed in.

        I.   Sample number
        II.   Project Number
        III.   Date
        IV.   Type of Samples
        V.   Department Code

SOP 04:   STANDARD OPERATING PROCEDURE FOR AIR SAMPLE PREPARATION

Reference: NIOSH Method 7400 (Issue 2, 15 August 1994) and EPA 40 CFR 763 (Vol. 51, No. 119)

Materials:

Acetone
Triacetin
Pipette (for triacetin)
Quick Fix (or appropriate subsitute) with syringe
Exacto Knife with #22 curved blade
Forceps
Lint-free lens tissues
75 X 25 mm glass microscope slides
25 X 25-mm glass cover slips
Slide trays
Water bottle
Paper towels
Felt tip pen (Sharpie)

Procedure:

1.   Turn on Quick Fix and wait for Quick Fix to heat up.
2.   Put acetone into syringe associated with Quick Fix. Insert syringe into Quick Fix.
3.   Obtain a microscope slide and write the project code number and sample I.D. at the top of the slide. Write the client name on the bottom of the first and last slide of the group.
4.   Open cassette carefully making certain the filter and support were placed in the cassette correctly during sampling.
5.   Cut a 1/4 section of filter with knife using a rocking motion. This is performed while filter is on the support pad. A 1/4 section is cut if standard 25-mm cassettes are used. Cut a 1/8 section if 37-mm cassettes are used.
6.   Close cassette with remaining 3/4 filter in place.
7.   Wipe Premark slide with a lint free tissue.
8.   Pick up the 1/4 section of the cut filter on its edge with the forceps and place the filter in the middle of the slide with the dust side up.
9.   Place slide into Quick Fix and apply 2/10 ml of acetone with syringe. Wait approximately five to ten seconds for the filter to dissolve.
10.   Take the slide out and apply one drop of triacetin to the clear filter.
11.   Wipe a cover slip with lint-free tissue and set the cover slip over the filter.
12.   Mark edge of filter on underside of slide with red felt tip pen.
13.   Place prepared slide in slide tray and repeat the procedure for all other air samples in the group.

14. Clean all equipment between each sample preparation.
15. Archive remaining 3/4 section of filter (in cassette) by SCN or PCN and client name.

SOP 05: **STANDARD OPERATING PROCEDURE FOR AIR SAMPLE ANALYSIS**

Reference: NIOSH 7400 Method, Issue 2 15 August 1994.

Equipment:
Phase Contrast Microscope with Walton-Beckett graticule
Hand-held counter
HSE/NPL test slide

Procedure:
1. Calibration is performed by following the manufacturer's instructions, the procedures stated in the NIOSH 7400 Method and EPA's 40 CFR, Vol. 51, No. 119 (see Appendix D).
   a) Adjust light source for even illumination (use Kohler illumination when possible). Focus on particulate on filter. Make sure field iris is centered on sample and is in focus.
   b) Check phase-shift detection limit using HSE/NPL test slide.
2. Fiber count is achieved using NIOSH 7400 Method ("A" counting rules). The 10 fibers per 100 fields lower detection limit is retained from P & CAM 239. Rules below are from NIOSH 7400 Method:
   a) Count only fibers longer than 5 um. Measure length of curved fibers along the curve on the graticule.
   b) Count only fibers with a length-to-width ratio greater than or equal to 3 to 1.
   c) For fibers which cross the boundary of the graticule field:
      i. Count any fiber longer than 5 um which lies entirely within the graticule area.
      ii. Count as 1/2 fiber any fiber with only one end lying within the graticule area provided that the fiber meets rules (a) and (b).
      iii. Do not count any fiber that crosses the graticule boundary more than once.
      iv. Reject and do not count all other fibers.
   d) Count bundles of fibers as one fiber unless observing both ends of a fiber can identify individual fibers.
   e) Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 fields regardless of count.

3. Hand-held counters are used, in most cases, to keep track of the number of fields counted on a filter. When the total fields necessary for completion have been counted, all data can then be entered on the analyst's bench sheet.

4. Calculations (from 40 CFR 763 Vol. 51, No. 119)
   a) If no volume is submitted, then fiber density E (fibers/mm^2), is calculated by dividing the total fiber count, F: minus the mean field

blank count, B: by the number of fields, N: and the field area, A_f, (0.00785 mm^2):

$$E = \frac{F - B}{(n)(A_f)} = \text{fibers/mm2}$$

b) When a volume is submitted, then fiber concentrations, C (fibers/cubic centimeter), is calculated using, E, from above multiplied by the effective collection of area of the filter, A_c (385 mm^2 for a 25 mm filter), divided by the volume, V (liters):

$$C = \frac{(E)(A_c)}{V(10^3)} = \text{fibers/cc}$$

5. Follow all QC procedures for air sample analysis.

SOP 06:   STANDARD OPERATING PROCEDURES FOR AIR SAMPLE ANALYSIS REPORTS

Rules for Text Report for Air Samples

(1)   This report is used for air samples received from a client and analyzed by BADLANDS.

   (a)   Cover Page: All information should be centered.

   i)   Full name and address of the client should be typed in, including zip code.

   ii)   Full project name will also be typed and centered along with the client's project number, if one.   There should be two spaces between the colon and the project name.

   iii)   PCN is the BADLANDS assigned project #.

   iv)   The date is the month/year the samples were analyzed.

   (b)   Description/Information Page:

   i)   Client name should be the same as listed on the cover page.

   ii)   Project location is the same as the project name listed on the cover page.   List the project number also if there is one.

   iii)   The representative is the client contact for the project.   If there is not a client contact listed on the job description sheet, look on the chain of custody.   **(The contact is not the sampler.)**   If there is no contact listed, leave this blank.

   iv)   Description of Work and Laboratory should always be the same.

   v)   BADLANDS Representative(s): The person who analyzed the samples should be listed.   If more than one analyst is listed on the data sheets, then each person should be listed.

   (c)   Text: Remains the same except under the personnel paragraph the name(s) of the analysts need to be filled in.   All the analysts need to complete the NIOSH 582, therefore only the names will change, never the text.

Air Sample Data Sheet

(2)   Document Program Set-up: The program is set-up to allow the user to enter data by using the mouse, pre-programmed keys, and the use of arrow keys.

      (a)   Data for the air samples is in two sections; the numeric data and the description text data. Type the samples in the order that they appear on the chain of custody. Always type the exact information from the chain of custody. If you have a question, ask the lab manager or the office manager. Page, client name, project, project code number, date and analyst information should be entered first. Using the 'client' key and mouse or just the mouse can do this.

            i)   The number of pages is in the top right hand corner of the data sheet. Determine the number of pages by the number of samples submitted.

            ii)   The client name should be the same as listed on the cover page of the text report. There should be two spaces after the colon.

            iii)   The PCN is the BADLANDS project code number. There should be two spaces after the colon.

            iv)   The project name should be exactly the same as listed on the cover page of the text report. There should be two spaces after the colon.

            v)   The date is the day the analysis was done. This can be found on the the written result sheet. Each page may be different.

            vi)   On each lab data form, the analyst is listed. Check which analyst(s) should be listed for the ten samples you just typed and list them on the left lower corner under "analyst." If there was more than one analyst list them as name / name.

      (b)   Make sure all of your pages are in correct order.

            i)   Sample #: numbers should always follow the order on the bench sheets. The sample number given by the client should be placed in the client number column in the same order.

            ii)   Sample date: the date should be entered as *mm/dd/yy*. The date should be on the chain of custody. If one is not listed, leave the cell blank.

iii)   Start and Stop time: the column for time says "24 hour clock," but do not change the time to a "24 hour" term if it is not listed as such on the chain of custody. Always type in what is on the chain of custody.

iv)   Total minutes: if there isn't a total listed on the chain of custody, leave it blank.

v)   Flow rate: sometimes only an average is listed under this area. Do not fill in the start and stop times if they are blank on the chain of custody - only the average flow rate.

vi)   Air volume liters: this is listed on the chain of custody and the lab sheet.

vii)   Fbrs/Flds: this number is located on the lab sheet.

viii)   LOQ: this number is located on the lab sheet. Sometimes the symbol <LOQ is on the sheet. Type it in with the LOQ in caps. EXCEPTION: If the report is for Virga Environmental Services do not use the <LOQ. You must use a numeric entry. If the LOQ is 0.020 then you must type <0.020.

ix)   Description data: the same samples should be listed, in the same order and the same location as above (the numbers will be entered automatically on this program, but double check that the numbers correspond correctly). Type in the data as shown on the chain of custody under Location/Description. Use commas to separate information.

x)   VOIDS: If a sample is void, you must type in the information that is on the chain of custody anyway, leaving cells blank if no information is listed. In the Fbrs/Flds, LOQ and F/cc cells you must type VOID in caps. An LOQ however will be listed for voided samples, which have an air volume, therefore you, will type VOID only in the Fbrs/Flds and the F/cc cells. In the area for the description sample data, type in the information listed on the chain of custody and follow it with "VOID: AND THE REASON" in capital letters.

xi)   Field Blanks: Samples that are field blanks will have data in the sample number, date and Fbrs/Flds cells only. In the descriptive data area type "Field Blank" with caps on the first letter only of each word.

xii)   Double check that the correct analyst and page number is listed before you go to the next page.

SOP 07: STANDARD OPERATING PROCEDURES FOR QUALITY CONTROL FIBER COUNTING ON AIR FILTERS (PCM)

It is the policy of Badlands, to be in compliance with the OSHA, Section 1926.58, Appendix A requirements for asbestos fiber counting when personal samples are analyzed. All submitted samples for fiber counting will be subject to the following requirements that are consistent with OSHA requirements:

1.   All microscopes equipment is calibrated weekly with an HSE/NPL phase contrast test slide. Calibrations are recorded in a logbook.

2.   Badlands analysts are appropriately trained for each type of analysis.

3.   BADLANDS participates in the AIHA AAR/AAT program.

4.   BADLANDS participates in a voluntary round robin QA/QC program on a quarterly basis with several other laboratories throughout the United States.

5.   All PAT and voluntary round robin QA/QC results are posted in the laboratory.

6.   Ten percent (10%) of all submitted samples are re-analyzed (replicate or duplicate) on a blind basis. This includes submitted blanks.

7.   Quality control slides, consisting of routine field samples and PAT samples, are counted on a blind basis by each microscopist at a frequency of one per day.

8.   All analysts who perform PCM analysis are required to attend the NIOSH 582 Training Course, or its equivalent.

9.   All quality control information is recorded in the QC Log Book.

10.   All air sample reports are calculated with blank corrections and checked and reviewed three times.

11.   In-house Alphabet slides are read every three (3) months.

Procedures for Avoiding Contamination:

1.   Samples

- Reject all cassettes found opened in shipping or transit
- Each sample is prepared individually under a high efficiency hood
- All archived samples are kept in their original cassettes

2.   Slides and Cover Slips

- Never re-use slides or cover slips
- Wipe with lint-free lens tissue before using

3. Exacto Knives, forceps, etc.:
- Clean after every sample preparation with water and paper towel

4. Hoods:
- Wipe utilized utensils between each sample preparation
- Change pre-filters as necessary
- Change filters as necessary

5. Acetone:
- Close bottle immediately after syringe is filled
- Insert syringe directly into Quick Fix

6. Triacetin and Applicator:
- Never touch pipette to filter
- Pipette is discarded when empty and a new one is then filled
- Close bottle immediately after pipette is filled

SOP 08: STANDARD OPERATING PROCEDURES FOR CORRECTIVE ACTIONS

In cases where data QC fails to meet the required limits, or the overall QA of the project is suspect, the QAM will be notified. Corrective actions shall be determined based on the severity of the breakdown and the requirements of the customer(s).

In general, QC failures in precision and/or accuracy will require repeat measurements and/or sample preparation. Deficiency of the QA plan will be dealt with on a project by project basis depending on the severity of the problem and the requirements of the customer.

In all cases, the QAM or the Lab Supervisor in conjunction will make the final decision in QA/QC failure or breakdown with the Supervisor (s) whose date is impacted.

Following the procedures and protocols set forth in this document can minimize the need for corrective action.

Corrective Actions for Air Sample Analysis:

1. When a blind recount comparison is deemed unacceptable using the following formula:

    $\bar{x}$ = average of counts
    C.v. = 0.35 (in-house coefficient of variation)
    $z = 2.77 \ (x) \ (C.v.)$
    Δx = absolute difference between counts
    If $\Delta x < z$ then acceptable
    If $\Delta x > z$ then not acceptable

    All remaining samples in the set are reanalyzed and a new comparability test will be performed for each sample. If any of the tests are found to be unacceptable the sample in question is reanalyzed by one or more different analysts to determine an accurate count.

2. If counts from a field sample are found to be unacceptable after being reanalyzed in the laboratory an assessment is made of the field conditions in an attempt to alleviate the deviation. If no solution is found all remaining samples from that project are analyzed in the laboratory.

Non-Conformances:

Non-conformances may include: 1) instrument failures/problems; 2) incomplete/missing sample documentation; 3) unacceptable sample condition; 4) exceeding sample holding times; 5) improper sample storage; 6) incorrect sample

preparation; 7) wrong analysis method/procedure; 8) improper field sampling methods; 9) QC data outside acceptance limits; 10) calibration requirements not met; 11) data recording, transcription or validation errors; and 12) improper reporting.

In all cases, a nonconformance report shall be made giving a description of the problem, the corrective action taken, the name of the individual reporting the problem, the date the problem was discovered, and the affected project or samples.

All employees shall be responsible for reporting any nonconformance that they observe or identify it to the QC Coordinator. The appropriate supervisor is responsible for assuring that the corrective actions are taken. Corrective actions may include: 1) re-calibrating instruments; 2) re-analyzing samples; 3) repairing instruments; 4) additional training of personnel; 5) substituting new lots of materials for defective materials; and 6) notifying clients or field personnel of missing paperwork, broken containers, or incorrect sampling methods or media.

SOP 09: STANDARD OPERATING PROCEDURES FOR INTERLABORATORY AIR SAMPLE ANALYSIS QUALITY CONTROL

<u>Materials:</u>   PCM Microscope
Data reporting sheet
Hand counters
26 prepared slides, from field and laboratory

<u>Procedure:</u>

1. Quality Control samples will be available every three months and will be analyzed in that time period.
2. Slides will be analyzed using the NIOSH 7400 method.
3. The QA/QC manager with results of other analysts will tabulate individual results. Overall results will be recorded and kept in the QC files.
4. Analysts will receive a Quality Control Summary showing where they fall in comparison with other analysts.
5. Individuals not falling within one standard deviation of the mean on any sample analysis will be asked to reanalyze that sample. Repeated deficiencies will be brought to the attention of the analyst's immediate supervisor.

SOP 10: STANDARD OPERATING PROCEDURE FOR MONTHLY/QUARTERLY SUMMARIES

Procedure: Summaries will be completed quarterly for air sample analysis. These include:

Air Sample Summaries

1) Analysis Total
2) Total QC
3) Daily Reference Slides
4) Results from Intra and Inter Laboratory Programs, (e.g. Round Robins and AAR)
6) Accuracy and Precision Tables and Graphs
7) Corrective Actions Taken During the Quarterly Period.

All summaries will be submitted to management for approval.

SOP 11:   STANDARD OPERATING PROCEDURE FOR PREPARATION HOODS

Quality Assurance

Sample preparation hoods are only utilized when the project field office is located at the Badlands corporate headquarters.

All utilized utensils are wet wiped between each sample preparation. Hoods are wet wiped inside and out at the end of every day. Hood pre-filters are replaced as necessary. The pre-filter is wet down with a spray bottle and carefully pulled out and placed in a lined, hazardous waste can for proper disposal by a local abatement company. All filters are replaced as necessary. The filter is wet down while in place. A large plastic bag is placed under housing and the filter is slowly lowered into the bag. The old filter and bag is then double bagged and turned over to a local abatement firm for safe disposal. All filter and pre-filter records are kept in the Quality Assurance logbook.

> Hood A: Sample Prep Hood with 12" x 12" HEPA Filter
> Made by: Air filtronix
> Received and Placed in Service: 5/12/95

SOP 12:   STANDARD OPERATING PROCEDURES FOR LABORATORY AIR MONITORING SCHEDULE

Annually: A.   Ambient/background sampling (PCM) is performed to evaluate laboratory make-up air cleanliness.

B.   All results are evaluated and changes in sampling strategy are implemented.

C.   Annual meetings are held with all laboratory staff to discuss changes in program and any response actions that took place over the previous calendar year.

SOP 13:   STANDARD OPERATING PROCEDURE FOR PHASE CONTRAST MICROSCOPE CALIBRATION

References: NIOSH 7400 Method, Issue 2, 15 August 1994.
            Zeiss Operating Instruction Manual, VII
            Standard KF-2

Materials: Phase Contrast Microscope
            HSE/NPL Test Slide
            Telescoping Eyepiece
            Stage Micrometer
            Kimwipes or Lens paper

Procedure:

1.   Light source must be in focus and centered on the condenser iris or annular diaphragm.
2.   Place a prepared slide on the mechanical stage of the microscope with the center of the filter under the objective lens. Focus the microscope into the plane of the filter.
3.   The illuminator field iris must be in focus, centered on the sample, having only the field of view illuminated.
4.   The phase rings must be concentric (i.e., the light must be contained within the dark circle so that no light is scattered around the edges). Always check when microscope has been moved.
5.   The phase shift detection limit should be checked with a HSE/NPL test slide. (i.e., optics must resolve line sets 1 through 3 completely. Sets 4 and 5 must partially visible, with sets 6 and 7 being invisible. Microscopes not fitting these criteria should not be used for airborne fiber counting).
6.   Each eyepiece, objective, and graticule should be calibrated with a stage micrometer. If any of the three are replaced or repaired, calibration should be repeated.

Care and Cleaning

1.   Always cover instrument with a plastic dust cover when microscope is not in use.
2.   Keep eyepieces in microscope body at all times in order to prevent dust from falling on the internal optics.
3.   Store microscope in a clean, safe place when not in use.
4.   Clean lens surfaces with a pressurized air source, soft cleaning brush, or lens paper.
5.   Carefully wipe off oil or fingerprints with a small amount of xylene, alcohol, or optical cleaner.

<u>Documentation</u>

1. Document weekly any adjustment, cleaning, or service to instrument in logbook provided. (Laboratory)
2. Document, as above, each time microscope is moved. (Field)
3. All forms of documentation must include an initial of analyst, date, and time of calibration.

SOP 14:   STANDARD OPERATING PROCEDURES FOR INTERNAL AUDITS

Procedure: The Quality Control/Quality Assurance officer will conduct annual laboratory audits. Items covered during the audit include:

1)   Sample Maintenance
2)   Calibration
3)   Preventive Maintenance
4)   Analytical Methods
5)   Data Verification
6)   Record Management

Audits will consist of general overview of laboratory and procedures. Audits may be towards a specific method/procedure.

Results of the audit shall be discussed with the Laboratory Supervisor and kept on file along with any corrective actions taken.

SOP 15: STANDARD OPERATING PROCEDURE FOR ADOPTING, REVIEWING, AND REVISING SOP'S

Procedure: Standard operation procedures shall be developed and implemented for all routine, standardized and special/critical operations.

<u>Adopting</u>

Standard operating procedures are created each time a new operation/procedure/method is presented. It is added to the list of SOP's under its appropriate heading. Adoption of a new procedure must be checked and approved by the Laboratory Director, QA/QC monitor and the Laboratory Supervisor.

<u>Reviewing</u>

Standard operating procedures are updated and reissued as required and reevaluated as needed. SOP's will be reviewed more frequently when changes have been made to references or the procedures; to determine suitability for continued use.

<u>Revising</u>

Each time a Standard Operating Procedure is revised, the revision date and number will be changed. Management must approve/reject of new SOP's. Copies of old SOP's will be kept on file.

SOP 16: STANDARD OPERATING PROCEDURES FOR TRAINING NEW PCM ANALYSTS

SECTION 17

ORGANIZATIONAL CHART

SECTION 18

EQUIPMENT LIST

TABLE A
EQUIPMENT INVENTORY

Item	Manufacturer	Model#	Serial#	Major Components	Date Received
PCM Microscope	Zeiss (#1)	KF-2	002690	Phase Rings Objectives Oculars	August 1989
PCM Microscope	Zeiss (#2)	KF-2	002693	Phase Rings Objectives Oculars	August 1989

- Any failed, replaced or repaired equipment will be documented on the equipment replacement or repair form.

Item	Manufacturer	Model#	Serial#	Major Components	Date Received
Permamount	Birmingham Instruments	N/A	3JU88	N/A	N/A
Quick Fix	R.J. Lee Group Inc.	2112A	22306	N/A	N/A
HEPA Hood (Air)	Falcon Fabrication	FC550	N/A	HEPA Filter Pre-Filter Fans	May 1989

SOP No. SP-2
Revision: #4
Date: 1/96
Page 1 of 2
Approved by: QAM
___Supervisor

Badlands

Equipment Calibration
Schedule Laboratory

Item	Calibration Schedule	Laboratory Personnel	Reference Document	Acceptance Criteria
Zeiss PCM	Daily	PCM Analysts	S.O.P. QA-8 Zeiss Operating Manual KF-2	
Air Samples	Monthly	PCM Analysts	S.O.P. No QA-5	

SOP No. SP-2
Revision: #4
Date: 1/96
Page 1 of 2
Approved by: QAM
___Supervisor

SECTION 19

DISTRIBUTION RECORD

AIHA
The Essential Source

November 12, 2003

THOMAS D. KOCH
BADLANDS ENVIRONMENTAL CONSULTANTS INC.
1006 E. CENTRAL AVE.
UNIT A
BISMARCK ND 58501

Lab ID# 158228

Dear THOMAS D. KOCH

Enclosed is your Proficiency Analytical Testing (PAT) Round 155 results.

PAT Round 156 sample kits will be mailed to laboratories around January 1, 2004.
Results will be due to AIHA on February 1, 2004. The analytes for round 156 are:

> **Metals** - cadmium, chromium, lead
> **Organics** - methanol
> **Asbestos** - amosite

If you have any questions, please contact Keesha McCormick at AIHA, (703) 846-0797.

**Your Password to enter data via the internet is now included on the submission form included
with the PAT samples. Your password is in the upper left hand corner (next to your lab ID#) of the
mailing address label. Please do not call AIHA for your password. Because of security concerns,
passwords will not be given over the phone.**

Please note: After submitting your data on the PAT data web site, it is very important to print the
confirmation page. Save this page as verification that results have been submitted.

The address to enter PAT results is: http//www.aiha.org/pat

**The AIHA Laboratory Quality Assurance Programs, Policies and Application for AIHA
accreditation are available on-line.**
http://www.aiha.org

Note: The Policies for 2003 comply with ISO 17025.

The application covers the following programs:
1. Industrial Hygiene Laboratory Accreditation Program (IHLAP) including Bulk Asbestos as an analyte
2. Environmental Lead Laboratory Accreditation Program (ELLAP)
3. Asbestos Analysts Registry (AAR)
4. Environmental Microbiology Accreditation Program (EMLAP)

Sincerely,

[signature]

Keesha T. McCormick
PAT Data Specialist

138228

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
INDIVIDUAL LABORATORY REPORT FOR ROUND 155
LAB ID=138228   NOVEMBER 11, 2003
BADLANDS ENVIRONMENTAL CONSULTANTS, INC., BISMARCK, ND 58501

CONTAMINANT (ABV.)	UNIT	SAMPLE NO.	REPORTED RESULTS	MEAN VALUES *	ACCEPTABLE RANGE# LOWER	UPPER	Z # SCORE	LAB PERFORMANCE
ASBESTOS/FIBERS (ASB/ANF)	(F/MM2)	1	97.0000	125.6590	61.5729	212.3637	-1.21	A
	(F/MM2)	2	294.0000	239.6501	17.4286	405.3087	-0.30	A
	(F/MM2)	3	201.0000	160.7304	78.7579	271.6364	1.18	A
	(F/MM2)	4	85.5000	70.3428	38.3880	132.3994	0.45	A

* Mean values are the mean of all laboratories based on original scales except for asbestos.
  Asbestos results are calculated based on transformed data. Therefore, asbestos performance limits are
  not symmetrical to the mean value.
Upper limit: mean value + 3 standard deviations
  Lower limit: mean value - 3 standard deviations
& Z Score = (reported result-mean value)/standard deviation
@ A: Analysis acceptable   -: Results not reported
  H: Results > upper limit (Z > 3), not acceptable   L: Results < lower limit (Z < -3), not acceptable
Note: the acceptability of reported results is based on z-scores. This is why a reported result may appear
  acceptable according to performance limits, but be identified as an outlier.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
LABORATORY YEAR-TO-DATE PERFORMANCE REPORT FOR ROUND 155
LAB ID=158228 NOVEMBER 11, 2003
BADLANDS ENVIRONMENTAL CONSULTANTS, INC., BISMARCK, ND 58501

SAMPLE TYPE	ROUND	ROUND * PERFORMANCE	ACCUMULATED PERFORMANCE 4 ROUNDS (%)		2 ROUNDS (%)		PROFICIENCY RATING #
ASBESTOS/FIBERS	152	4/4					
	153	4/4					
	154	4/4					
	155	4/4	16/16	100	8/8	100	P

* The denominators represent the total number of samples to be analyzed.
  The numerators represent the number of acceptable results.
  A '-' represents non-submittal and is considered as a zero in the numerator.
P : Proficient   N: Nonproficient
  Performance ratings are based on expected results over four rounds (one year). A lab's
  performance on each sample type is rated proficient (P), if: 1) three-fourths   (75%) or more
  of the accumulated results over four rounds are acceptable or 2) for the   last two rounds,
  all samples are analyzed and the results are 100% acceptable. If a   laboratory receives
  samples for a contaminant and does not report the data, the results will be   considered
  unacceptable for that contaminant.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
SUMMARY RESULTS OF ALL LABS FOR ROUND 155

CONTAMINANT (UNIT)	SAMPLE NO.	SUMMARY STATISTICS OF ALL LABS				# LABS RATED	# OF LABS ACCEPTABLE	# LOW OUTLIER	# HIGH OUTLIER
		N	MEAN*	STD@	RSD&				
CADMIUM (MG)	1	229	0.0089	0.000168	4.3	229	213	10	6
	2	229	0.0116	0.000465	4.0	229	219	7	3
	3	229	0.0154	0.000618	4.0	229	216	9	4
	4	229	0.0077	0.000307	4.0	229	215	9	5
LEAD (MG)	1	232	0.0898	0.003505	4.0	232	223	4	5
	2	232	0.0294	0.001177	4.0	232	213	10	9
	3	232	0.0585	0.002359	4.0	232	220	7	5
	4	232	0.1165	0.00466	4.0	232	218	7	7
ZINC (MG)	1	229	0.1002	0.004374	4.1	229	216	7	6
	2	229	0.1959	0.007836	4.0	229	213	10	6
	3	229	0.0499	0.002144	4.3	229	208	10	11
	4	229	0.1475	0.0059	4.0	229	207	12	10
SILICA (MG)	1	67	0.0503	0.008839	17.6	67	63	1	3
	2	67	0.0995	0.015089	15.2	67	64	2	1
	3	67	0.1083	0.016489	15.0	67	66	1	0
	4	67	0.0701	0.011388	16.2	67	65	0	2
ASBESTOS/FIBERS (F/MM2) +	1	164	125.6590	25.1318	20.0	842	696	74	72
	2	164	239.6001	47.93002	20.0	842	693	120	29
	3	164	160.7854	32.14608	20.0	842	720	89	33
	4	164	78.3428	15.66857	20.0	842	773	27	42
CHLOROFORM (MG)	1	196	0.2995	0.012328	4.1	196	182	7	7
	2	196	0.4942	0.01977	4.0	196	185	5	6
	3	196	1.1335	0.045341	4.0	196	182	6	8
	4	196	0.1013	0.005991	5.9	196	181	6	9
1,2-DICHLOROETHANE (MG)	1	196	1.2324	0.049297	4.0	196	186	6	4
	2	196	0.9142	0.036729	4.0	196	190	3	3
	3	196	0.3915	0.015661	4.0	196	188	2	6
	4	196	0.2050	0.008324	4.1	196	185	5	8
TRICHLOROETHYLENE (MG)	1	197	0.8776	0.035104	4.0	197	187	4	4
	2	197	1.1292	0.045166	4.0	197	186	7	4
	3	197	0.2063	0.011851	4.0	197	183	7	7
	4	197	0.4880	0.019553	4.0	197	185	4	6

* MEAN - The mean of all laboratories. These values were
listed on the individual laboratory report.
@ STD - standard deviation
& RSD - relative standard deviation=((STD/MEAN)*100%)
+ - Results for fibers are calculated on transformed data.

AIHA
The Essential Source

August 27, 2003

THOMAS D. KOCH
BADLANDS ENVIRONMENTAL CONSULTANTS INC.
1006 E. CENTRAL AVE.
UNIT A
BISMARCK ND 58501

Lab ID# 158228

Dear THOMAS D. KOCH

Enclosed are your Proficiency Analytical Testing (PAT) Round 154 results.

PAT Round 155 sample kits will be mailed to laboratories around October 1, 2003.
Results will be due to AIHA on November 1, 2003. The analytes for round 155 are:

Metals   - cadmium, lead, zinc
Organics - methanol (MOH)
Asbestos - chrysotile

If you have any questions, please contact Keesha McCormick at AIHA, (703) 846-0797.

Your Password to enter data via the Internet is now included on the submission form included
with the PAT samples. Your password is in the upper left hand corner (next to your lab ID#) of the
mailing address label. Please do not call AIHA for your password. Because of security concerns,
passwords will not be given over the phone.

Please note: After submitting your data on the PAT data web site, it is very important to print the
confirmation page. Save this page as verification that results have been submitted.

The address to enter PAT results is: http://www.aiha.org/pat

**The AIHA Laboratory Quality Assurance Programs, Policies and Application for AIHA
accreditation are available on-line.**
http://www.aiha.org

Note: The Policies for 2002 comply with ISO 17025.

The application covers the following programs:
1. Industrial Hygiene Laboratory Accreditation Program (IHLAP) including Bulk Asbestos as an analyte
2. Environmental Lead Laboratory Accreditation Program (ELLAP)
3. Asbestos Analysts Registry (AAR)
4. Environmental Microbiology Accreditation Program (EMLAP)

Sincerely,

[signature]

Keesha T. McCormick
PAT Data Specialist

American Industrial Hygiene Association
2700 Prosperity Ave., Suite 250, Fairfax, VA 22031
(703) 849-8888 ;(703) 207-8558 fax
InfoFax Service Line (703) 641-INFO or Internet: infonet@aiha.org

156228

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
INDIVIDUAL LABORATORY REPORT FOR ROUND 154
LAB ID=156228   AUGUST 26, 2003
BADLANDS ENVIRONMENTAL CONSULTANTS, INC., BISMARCK, ND 58501

CONTAMINANT (ABV.)	UNIT	SAMPLE NO.	REPORTED RESULTS	MEAN VALUES *	ACCEPTABLE RANGE# LOWER	UPPER	Z & SCORE	LAB & PERFORMANCE
ASBESTOS/FIBERS (ASB/PNF)	(F/MM2)	1	175.7000	222.9430	117.3000	362.2187	-1.23	A
	(F/MM2)	2	155.9000	167.3313	80.1133	281.2571	-0.36	A
	(F/MM2)	3	197.5000	252.3775	135.6588	405.0368	-1.30	A
	(F/MM2)	4	69.6000	86.6302	42.4488	146.4050	-1.04	A

* Mean values are the mean of all laboratories based on original scales except for asbestos.
  Asbestos results are calculated based on transformed data. Therefore, asbestos performance limits are not symmetrical to the mean value.
Upper limit: mean value + 3 standard deviations
  Lower limit: mean value - 3 standard deviations
& Z Score = (reported result-mean value)/standard deviation
& A: Analysis acceptable   -: Results not reported
  H: Results > upper limit (z > 3), not acceptable   L: Results < lower limit (z < -3), not acceptable
note: the acceptability of reported results is based on z-scores. This is why a reported result may appear acceptable according to performance limits, but be identified as an outlier.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
LABORATORY YEAR-TO-DATE PERFORMANCE REPORT FOR ROUND 154
LAB ID=150228 AUGUST 26, 2005
BADLANDS ENVIRONMENTAL CONSULTANTS, INC., BISMARCK, ND 58501

| SAMPLE TYPE | ROUND | ROUND * PERFORMANCE | ACCUMULATED PERFORMANCE | | PROFICIENCY RATING # |
			4 ROUNDS (%)	2 ROUNDS (%)	
ASBESTOS/FIBERS	151	4/4			
	152	4/4			
	153	4/4			
	154	4/4	16/16 100	8/8 100	P

* The denominators represent the total number of samples to be analyzed.
  The numerators represent the number of acceptable results.
  A '-' represents non-submittal and is calculated as a zero in the numerator.
P : Proficient   &: Nonproficient
  Performance ratings are based on expected results over four rounds (one year). A lab's
  performance on each sample type is rated proficient (P), if: 1) three-fourths (75%) or more
  of the accumulated results over four rounds are acceptable or 2) for the last two rounds,
  all samples are analyzed and the results are 100% acceptable. If a laboratory receives
  samples for a contaminant and does not report the data, the results will be considered
  unacceptable for that contaminant.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
SUMMARY RESULTS OF ALL LABS FOR ROUND 154

CONTAMINANT (UNIT)	SAMPLE NO.	SUMMARY STATISTICS OF ALL LABS				# LABS RATED	# OF LABS ACCEPTABLE	# LOW OUTLIER	# HIGH OUTLIER
		N	MEAN*	STD@	RSD(%)&				
CADMIUM (MG)	1	233	0.0153	0.000611	4.0	233	219	7	7
	2	233	0.0021	0.000107	5.1	233	217	8	8
	3	233	0.0096	0.000363	4.0	233	223	5	5
	4	233	0.0099	0.000294	4.0	233	224	3	6
CHROMIUM (MG)	1	233	0.0808	0.004571	5.7	233	219	5	9
	2	233	0.1715	0.010061	5.9	233	222	7	4
	3	233	0.1204	0.006904	5.7	233	223	6	4
	4	233	0.0206	0.001132	5.5	233	216	7	10
LEAD (MG)	1	234	0.1465	0.005835	4.0	234	223	7	4
	2	234	0.0977	0.003907	4.0	234	222	6	6
	3	234	0.0775	0.0031	4.0	234	222	7	5
	4	234	0.3393	0.001671	4.8	234	219	5	10
SILICA (MG)	1	68	0.1119	0.016641	14.9	68	65	1	2
	2	68	0.0704	0.011429	16.2	68	66	1	1
	3	68	0.0865	0.013209	15.4	68	54	3	1
	4	68	0.1121	0.016664	14.9	68	64	2	2
ASBESTOS/FIBERS (F/MM2) +	1	164	222.9430	40.61919	18.3	823	769	15	39
	2	164	167.5313	33.02043	19.7	823	774	8	41
	3	164	252.5775	44.89634	17.8	823	766	20	37
	4	164	86.6302	17.32804	20.0	823	760	32	31
BENZENE (MG)	1	204	0.0605	0.003085	5.1	204	173	17	14
	2	204	0.1196	0.004856	4.1	204	185	7	12
	3	204	0.5003	0.02001	4.0	204	189	7	8
	4	204	0.2497	0.009988	4.0	204	190	4	10
O-XYLENE (MG)	1	204	0.9015	0.03604	4.0	204	190	9	5
	2	204	0.6854	0.027475	4.0	204	185	11	7
	3	204	1.2704	0.050817	4.0	204	187	9	8
	4	204	0.6839	0.020147	4.2	204	190	9	5
TOLUENE (MG)	1	204	0.8876	0.035506	4.0	204	197	4	3
	2	204	0.3900	0.015599	4.0	204	189	7	8
	3	204	1.6735	0.058908	4.0	204	184	12	8
	4	204	1.2043	0.048173	4.0	204	188	12	4

* MEAN - The mean of all laboratories. These values were
        listed on the individual laboratory report.
@ STD - standard deviation
& RSD - relative standard deviation((STD/MEAN)*100%)
+   - Results for fibers are calculated on transformed data.

![AIHA logo]

AIHA
The Essential Source

May 29, 2003

THOMAS D. KOCH
BADLANDS ENVIRONMENTAL CONSULTANTS INC.
1006 E. CENTRAL AVE.
UNIT A
BISMARCK ND 58501

Lab ID# 158228

Dear THOMAS D. KOCH

Enclosed are your Proficiency Analytical Testing (PAT) Round 153 results.

PAT Round 154 sample kits will be mailed to laboratories around July 1, 2003.
Results will be due to AIHA on August 1, 2003. The analytes for round 154 are:

> Metals   - cadmium, chromium, lead
> Organics - benzene (BNZ), o-xylene (OXY), toluene (TOL)
> Asbestos - amosite

If you have any questions, please contact Keesha McCormick at AIHA, (703) 846-0797.

Your Password to enter data via the Internet is now included on the submission form included
with the PAT samples. Your password is in the upper left hand corner (next to your lab ID#) of the
mailing address label. Please do not call AIHA for your password. Because of security concerns,
passwords will not be given over the phone.

Please note: After submitting your data on the PAT data web site, it is very important to print the
confirmation page. Save this page as verification that results have been submitted.

The address to enter PAT results is: http://www.aiha.org/pat

The AIHA Laboratory Quality Assurance Programs, Policies and Application for AIHA accreditation
are available on-line.
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Note: The Policies for 2002 comply with ISO 17025.

The application covers the following programs:
1. Industrial Hygiene Laboratory Accreditation Program (IHLAP) including Bulk Asbestos as an analyte
2. Environmental Lead Laboratory Accreditation Program (ELLAP)
3. Asbestos Analysts Registry (AAR)
4. Environmental Microbiology Accreditation Program (EMLAP)

Sincerely,

Keesha T. McCormick
PAT Data Specialist

American Industrial Hygiene Association
2700 Prosperity Ave., Suite 250, Fairfax, VA
22031
(703) 849-8888 (703) 207-8558 Fax
InfoFax Service Line (703) 641-INFO or Internet:
infonet@aiha.org

154228

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
INDIVIDUAL LABORATORY REPORT FOR ROUND 153
LAB ID-154228   MAY 23, 2003
BADLANDS ENVIRONMENTAL CONSULTANTS, INC., BISMARCK, ND 58501

CONTAMINANT (ABV.)	UNIT	SAMPLE NO.	REPORTED RESULTS	MEAN VALUES *	ACCEPTABLE RANGE# LOWER	UPPER	Z & SCORE	LAB @ PERFORMANCE
ASBESTOS/FIBERS (ASB/MMF)	(F/MM2)	1	43.6000	59.5530	29.1810	100.6444	-1.42	A
	(F/MM2)	2	80.7000	115.3116	66.5027	194.8766	-1.63	A
	(F/MM2)	3	149.7000	160.7277	78.7546	271.6296	-0.35	A
	(F/MM2)	4	69.6000	71.8909	39.2285	121.4954	-0.16	A

* Mean values are the mean of all laboratories based on original scales except for asbestos.
  Asbestos results are calculated based on transformed data. Therefore, asbestos performance limits are
  not symmetrical to the mean value.
Upper limit: mean value + 3 standard deviations
  Lower limit: mean value - 3 standard deviations
& Z Score = (reported result-mean value)/standard deviation
@ A: Analysis acceptable   -: Results not reported
  U: Results > upper limit (Z > 3), not acceptable   L: Results < lower limit (Z < -3), not acceptable
Note: the acceptability of reported results is based on z-scores. This is why a reported result may appear
      acceptable according to performance limits, but be identified as an outlier.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
LABORATORY YEAR-TO-DATE PERFORMANCE REPORT FOR ROUND 153
LAB ID 150020 MAY 23, 2003
BADLANDS ENVIRONMENTAL CONSULTANTS, INC., BISMARCK, ND 58501

SAMPLE TYPE	ROUND	ROUND * PERFORMANCE	ACCUMULATED PERFORMANCE				PROFICIENCY RATING #
			4 ROUNDS (%)		2 ROUNDS (%)		
ASBESTOS/FIBERS	150	4/4					
	151	4/4					
	152	4/4					
	153	4/4	16/16	100	8/8	100	P

* The denominators represent the total number of samples to be analyzed.
  The numerators represent the number of acceptable results.
  A '-' represents non-submittal and is calculated as a zero in the numerator.
P : Proficient   N: Nonproficient
  Performance ratings are based on expected results over four rounds (one year).   A lab's
  performance on each sample type is rated proficient (P), if: 1) three-fourths   (75%) or more
  of the accumulated results over four rounds are acceptable or 2) for the   last two rounds,
  all samples are analyzed and the results are 100% acceptable. If a   laboratory receives
  samples for a contaminant and does not report the data, the results will be   considered
  unacceptable for that contaminant.

PROFICIENCY ANALYTICAL TESTING (PAT) PROGRAM
SUMMARY RESULTS OF ALL LABS FOR ROUND 133

CONTAMINANT (UNIT)	SAMPLE NO.	SUMMARY STATISTICS OF ALL LABS				# LABS RATED	# OF LABS ACCEPTABLE	# LOW OUTLIER	# HIGH OUTLIER
		N	MEAN	STD	RSD(%)				
CHROMIUM (MG)	1	234	0.0078	0.000311	4.0	234	220	9	5
	2	234	0.0148	0.000592	4.0	234	219	8	7
	3	234	0.0060	0.000206	4.1	234	217	8	9
	4	234	0.0099	0.000394	4.0	234	222	6	6
LEAD (MG)	1	235	0.0502	0.002367	4.0	235	226	5	4
	2	235	0.0197	0.000836	4.2	235	219	6	10
	3	235	0.0091	0.003166	4.3	235	227	7	1
	4	235	0.0492	0.00197	4.0	235	222	8	5
ZINC (MG)	1	234	0.1186	0.004775	4.0	234	212	14	8
	2	234	0.0604	0.002549	4.2	234	206	13	15
	3	234	0.1409	0.005935	4.0	234	209	16	9
	4	234	0.0983	0.004004	4.1	234	213	12	9
SILICA (MG)	1	69	0.1131	0.016787	14.8	69	65	2	2
	2	69	0.1540	0.021865	14.2	69	67	2	0
	3	69	0.0627	0.010452	16.7	69	65	2	2
	4	69	0.0880	0.013688	15.5	69	66	1	2
ASBESTOS/FIBERS (F/MM2) +	1	166	59.5530	11.91061	20.0	834	662	69	103
	2	166	115.3116	23.06252	20.0	834	705	74	55
	3	166	160.7277	32.14063	20.0	834	695	94	45
	4	166	7.8809	14.37818	20.0	834	747	37	50
METHYL ETHYL KETONE (MG)	1	200	0.1921	0.011522	6.0	200	185	5	10
	2	200	0.4871	0.025275	5.2	200	186	3	11
	3	200	0.9731	0.047653	4.9	200	185	7	8
	4	200	0.3635	0.019552	5.4	200	183	7	10
METHYL ISOBUTYL KETONE (MG)	1	200	0.7806	0.036043	4.6	200	182	9	9
	2	200	0.0985	0.006491	6.6	200	186	6	6
	3	200	0.2967	0.015193	5.1	200	186	7	7
	4	200	0.4937	0.023579	4.8	200	183	9	8

* MEAN - The mean of all laboratories. These values were
          listed on the individual laboratory report.
B STD - standard deviation
& RSD - relative standard deviation(STD/MEAN)*100%
+   - Results for fibers are calculated on transformed data.

APPENDIX 8

- **FIELD SAMPLING FORMS**
- **CONFIRMATION SOIL SAMPLE COLLECTION PROCEDURE (CSSCP-LIBBY-01)**
- **SAMPLE CUSTODY PROCEDURE (SCP-LIBBY-01)**
- **PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLE PROCEDURE (PSESP-LIBBY-01)**
- **FIELD LOGBOOK CONTENT AND CONTROL INFORMATION PROCEDURE (FLCCIP-LIBBY-01)**
- **PHOTOGRAPH DOCUMENTATION OF FIELD ACTIVITIES PROCEDURE (PDFAP-LIBBY-01)**
- **FIELD EQUIPMENT DECONTAMINATION PROCEDURE (FEDP-LIBBY-01)**

Date: <u>March 22, 2004</u>   Procedure No. <u>CSSCP-Libby-01</u>

Site: <u>Flyway Site</u>
      Libby, Montana

Confirmatory Soil Sample Collection Procedure (CSSCP)

The following procedure applies to the collection of confirmatory soil samples at the Flyway Site.

Equipment

<u>Sample Container</u> – the sample container will consist of quart-sized zip-top plastic bags.
<u>Trowel</u> – for collecting surface soil samples.
<u>Stainless Steel Mixing Bowl</u> – used to mix and homogenize composite soil samples after collection.
<u>Gloves</u> – for personnel protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.
<u>Field Clothing and Personal Protective Equipment (PPE)</u> – as specified in the health and safety plan (HASP).
<u>Field Sprayers</u> – will be used for decontaminating non-disposable sampling equipment between samples.
<u>Silica Sand</u> – for field equipment blank quality control (QC) samples.
<u>Field Logbooks</u> – used to record progress of sampling effort, and to record any problems and field observations.
<u>Field Sample Data Sheet (FSDS)</u> – used to record soil sample information.
<u>Permanent Marking Pen</u> – used to label sample containers.
<u>Index ID Stickers</u> – used to label containers.
<u>Plastic Buckets</u> – used to wash nondisposable field equipment between samples.
<u>Trash Bag</u> – used to dispose of gloves.
<u>Cooler</u> – used to store samples while in the field.
<u>Chain of Custody Record</u> – for ensuring custody of soil samples until shipping.
<u>Custody Seals</u> – for ensuring custody of soil samples during shipping.

Sample Collection

Don the appropriate PPE as specified in the HASP. A new pair of plastic gloves are to be worn for each soil sample collection.

- Each 100 ft. x 100 ft. grid will be subdivided into 20 ft. x 20 ft. subgrids (see Figure 5-3 discussed in Section 5.3 of the SAP). A composite soil sample will be collected from five (5) adjacent subgrids. Samples of surface soil will be collected at the approximate center-point of each subgrid (1, 2, 3, etc.). Partial grids will be sampled and composited in five (5) aliquots or lesser units for areas without five (5) subgrids.

- The soil samples will be collected from a 0-2 inch depth interval using a decontaminated trowel or appropriate disposable sampling devise.

- Each of the soil sample locations will be located using GPS equipment

- A description of the soil samples will be recorded in the field log book.

- Soil field duplicate samples will be collected at a rate of 1 per 20 (5 percent) of the field samples. These samples will be independently collected with separate sampling equipment.

Record Keeping and Quality Control

A field logbook should be maintained by each individual that is collecting samples as described in the Field Logbook Content and Control Procedure (FLBC-Libby-01). The FLBC procedure will detail specific conditions, but at a minimum, the following should be collected:

- Date;
- Time;
- Sampler (person collecting the sample);
- Weather Conditions;
- PPE used;
- Locations of any samples that could not be acquired; and
- Descriptions of any deviations of the FLBC for each sample.

- Quality control samples will include:
    - field duplicates; and
    - equipment blank samples.

Decontamination

All soil sampling equipment must be decontaminated prior to reuse. Specific instructions on sample equipment decontamination are included in Field Equipment Decontamination Procedure (FSDP-Libby-01). In general, the procedure to decontaminate all soil sampling equipment is outlined below:

- Remove all gross contamination with a plastic brush;
- Use clean water and a plastic brush to wash each piece of equipment;
- Remove excess water present on the equipment by shaking;
- Use a paper towel to dry each piece of equipment;
- Wrap dried equipment in aluminum foil.
- Once a week all soil sampling equipment will be cleaned using Alconox and clean water.

Spent gloves, and PPE must be disposed or stored properly at the site.

Date: <u>March 22, 2004</u>　　　　　　Procedure No. <u>SC-Libby-01</u>

Site: <u>Flyway Site</u>
　　　Libby, Montana

Sample Custody

Due to the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained at the Libby site office.

Responsibilities

<u>Sampler</u> - The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

<u>Project Quality Assurance Coordinator (PQAC)</u> – The PQAC is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The PQAC is responsible for coordinating with the subcontractor laboratory to ensure that adequate information is recorded on the custody forms.

<u>Field Sample Custodian</u> – The field sample custodian, when designated by the PQAC, is responsible for accepting custody of samples from the sampler(s) and properly packing the samples to be shipped to the laboratory assigned to do the analyses. A field sample custodian is typically designated only for large and complex field efforts.

Required Supplies

- Chain-of-Custody records
- Custody seals
- Sample labels or tags
- Clear tape

Procedures

Chain-of-Custody

This procedure establishes a method for maintaining custody of samples through use of chain-of-custody record. This procedure will be followed for all samples collected or split samples accepted.

Field Custody

1. Collect only the number of samples needed to represent the media being sampled. As few people as possible should handle samples.
2. Complete sample labels or tags for each sample, using waterproof ink.

Transfer of Custody and Shipment

1. Complete a chain-of-custody record for all samples (see Figure 1 for an example of a chain-of-custody record.
2. The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures.
3. In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.
4. If samples are left unattended or a person refuses to sign, this must be documented and explained on the chain-of-custody record.
5. Samples should be properly packaged for shipment and dispatched to the appropriate laboratory for analysis. Each shipment must be accompanied with a separate chain-of-custody record.
6. Include a chain-of-custody record identifying its content in all shipments (refer to Figure 1). The original record will accompany the shipment, and the copies will be retained by the PQAC and, if applicable, distributed to appropriate sample coordinators. Freight bills will also be retained by the PQAC as part of the permanent documentation.

Completing Chain-of-Custody

The following procedure is to be used to fill out the chain-of-custody record.

1. Record project number.
2. Record PQAC for the project.
3. Record the name and address of the laboratory where samples are being shipped.
4. Enter the project name/location.
5. Record overnight courier's air bill number (if shipped overnight).
6. Record sample location number.
7. Record sample number.
8. Note media type (media) and reference number.
9. Note sample type.
10. Enter date of sample collection.

11. Enter time of sample collection in military time.
12. When required, enter the names or initials of the samplers next to the sample location number of the sample they collected.
13. List parameters for analysis and the number of containers submitted for analysis.
14. Sign the chain-of-custody record(s) in the space provided. All samplers must sign each record.
15. If sample tags are used, record the sample tag number in the "Remarks" column.
16. Record date shipped.
17. The originator checks information entered and then signs the form including the current date and time (military).
18. Send the top two copies with the samples to the laboratory; retain the third copy for the project files. Retain additional copies for the project file or distribute as required to the appropriate sample coordinators.
19. The laboratory sample custodian receiving the sample shipment checks the sample label information against the chain-of-custody record. Sample condition is checked and anything unusual is noted under "Remarks" on the chain-of-custody record. The laboratory custodian receiving custody signs in the adjacent "Received" on the chain-of-custody record. The laboratory custodian receiving custody signs the chain-of-custody form and keeps the copy.

Custody Seals

Custody seals must be placed on the shipping containers (e.g., picnic cooler) prior to shipment. The seal should be signed and dated by a field team member.

Sample Shipping

See the packaging and shipping of environmental samples procedures PSES-Libby-01.

Date: <u>March 22, 2004</u>   Procedure No. <u>PSES-Libby-01</u>

Site: <u>Flyway Site</u>
      Libby, Montana

Packaging and Shipping of Environmental Samples

This procedure applies to the packaging and shipping of all environmental samples (soil samples and air samples) specific to the SAP. No chemicals will be shipped with any of the soil samples.

Responsibilities

The Project Quality Assurance Coordinator (PQAC), is responsible for ensuring that packaging and sampling procedures are conducted in accordance with this procedure.

Required Equipment

- Coolers with return address
- Plastic Ziploc®-type bags, small and large
- Clear tape
- Duct tape
- Large heavy-duty plastic garbage bag
- Bubble wrap
- Custody seals
- Completed chain-of-custody record
- Completed bill of lading, if applicable
- "This End Up" and directional arrow labels

Procedures

The following steps must be followed when packaging samples.

1. Select a sturdy cooler in good repair.
2. Make sure that all the sample bags are secured.
3. Place the completed chain-of-custody record for the laboratory into a plastic zip-top bag, tape the bag to the inner side of the cooler lid, and close the cooler.
4. The cooler lid shall be secured with duct tape (or other similar type tape) by wrapping each end of the cooler a minimum of two times. Attach a completed chain-of-custody seal across the hinges of the cooler on opposite sides. The custody seals should be affixed to the cooler with half of the seal on the strapping

tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with clear tape over the custody seals.

5. The shipping container lid must be marked "THIS END UP" and arrow labels that indicate the proper upward position of the container should be affixed to the cooler. A label containing the name and address of the shipper shall be placed on the outside of the container. The name and address of the laboratory shall be placed on the container, or when shipped by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

Date: March 22, 2004   Procedure No. FLBC-Libby-01

Site: Flyway Site
        Libby, Montana

Field Logbook Content

A field logbook will be kept to document field sampling work efforts conducted at the Flyway Site.

1. Field logbooks will be bound with lined, consecutively numbered pages. All pages will be numbered prior to initial use of the log book. Prior to use in the field, each logbook will be marked with a specific document control number issued by the project quality assurance coordinator.

2. The following information will be recorded on the cover of the logbook.

   a. Field logbook document control number.
   b. Activity (if the logbook is to be activity-specific) and location.
   c. Start date.

3. Operation

   The following is a list of requirements that must be followed when using a logbook:

   a. Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook.
   b. Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made.
   c. Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion.
   d. Do not remove any pages from the logbook.

   Specific requirements for field logbook entries include:

   a. Initial and date each page.
   b. Sign and date the final page of entries for each day.
   c. Initial and date all changes.

d. A new author must sign and print his/her name before additional entries are made.

e. Draw a diagonal line through the remainder of the final page at the end of each day.

f. Record the following information on a daily basis:

- Date and time;
- Name of individual making entry;
- Names of field team and other persons on site;
- Description of activity being conducted including station or location (i.e., soil sample location, etc.);
- Weather conditions (i.e., temperature, etc.);
- Level of personal protection to be used;
- Serial numbers of instruments;
- Required calibration information; and
- Serial/tracking numbers on documentation (e.g., carrier air bills).

g. At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. A GPS location should be included for each sample location. All maps or sketches made in the logbook should have descriptions of the features shown.

h. Other events and observations that should be recorded include:

- Changes in weather that impact field activities;
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation;
- Problems, downtime, or delays; and
- Upgrade or downgrade of personal protection equipment.

4. Post-Operation

To guard against loss of data due to damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated, and that corrections were made properly. The completed logbook shall be submitted to the field office.

5. Restrictions/Limitations

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by Remedium Group, Inc. (a subsidiary of W.G. Grace & Co.) and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these notebooks should be factual, clear, precise, and non-subjective. Field logbooks, and entries within, are not to be utilized for personal use.

Date: <u>March 22, 2004</u>   Procedure No. <u>PDFAP-Libby-01</u>

Site: <u>Flyway Site</u>
      Libby, Montana

Photographic Documentation of Field Activities Procedure (PDFAP)

Photograph recordings made during field investigations are used as an aid in documenting and describing site features, sample collection activities, and equipment used.

The project quality assurance coordinator is responsible for ensuring that the format and content of photographic documentation are in accordance with this procedure.

The photographer shall seek direction from the project quality assurance coordinator and discuss the visual documentation requirements and schedule. The photographer is responsible for maintaining a logbook.

Required Equipment

- 35 mm camera, disposable single use camera (35mm or panoramic use) or digital camera
- Logbook
- Indelible black or blue ink pen
- Standard reference markers
- Medium speed, or multi purpose fine-grain, color, 35 mm film or storage medium for digital camera

Documentation

- A commercially available, bound logbook will be used to log and document photographic activities.

Operation

- The photographer should be prepared to make a variety of shots, from close-up to wide-angle.
- All still film photographs should be made using a medium speed, multi purpose fine-grain, color negative film in 35 mm format.
- No preference of digital storage medium is specified and is left to the discretion of the photographer.

Slate Information

- When directed by the project quality assurance coordinator, each new roll of film or digital storage medium shall contain upon the first usable frame (for film) a slate with consecutively assigned control numbers.

Caption Information

- All still photographs will have a full caption permanently attached to the back or permanently attached to a photo log sheet. The caption should contain the following information.

    - Film roll control number(if required) and photograph sequence number
    - Date and time
    - Description of activity/item shown
    - Direction (if applicable)
    - Photographer

    Digital media should be downloaded at least once each day.

Close-up and Feature Photography

When directed by the Project Quality Assurance Coordinator, close-up photographs should include a standard reference marker of appropriate size as an indication of the feature size and contain a slate marked with the site name and identifying label, such as a soil sample number, that clearly communicates to the viewer the specific feature being photographed.

Site Photography

Site photography, in general, will consist predominately of medium and wide-angle shots. A standard reference marker should be placed adjacent to the feature or, when this is not possible, within the same focal plane.

Panoramic

In situations where a wide-angle lens does not provide sufficient subject detail, a single use disposable panoramic camera is recommended.

Photographic Documentation

Photographic activities must be documented in a photographic logbook or in a section of the field logbook. The photographer will be responsible for making proper entries.

The following information should be maintained in the appropriate logbook:

- Photographer name;
- If required, an entry shall be made for each new roll/tape control cumber assigned;
- Sequential tracking number for each photograph taken (for digital cameras, the camera generated number may be used);
- Date and time (military time);
- Location;
- A description of the activity/item photographed;
- If needed, a description of the general setup, including approximate distance between the camera and the subject, may be recorded in the logbook;
- Record as much other information as possible to assist in the identification of the photographic document.

Post Operation

All film will be sent for development and printing to a photographic laboratory (to be determined by the photographer). The photographer will be responsible for arranging transport of the film from the field to the photographic laboratory. The photographer shall also be arranging delivery of the negatives and photographs, or digital storage medium to the project management representative.

Documentation

At the end of each day's photographic session, the photographer(s) will ensure that the appropriate logbook has been completely filled out and maintained.

Photographs and the associated set of negatives, digital media, and original unedited documentary videotape recording will be submitted to the project files and handled according to contact records requirements.

Completed pages of the appropriate logbook will be copied weekly and submitted to the project files

Date: <u>March 22, 2004</u>   Procedure No. <u>FEDP-Libby-01</u>

Site: <u>Flyway Site</u>
      Libby, Montana

Field Equipment Decontamination Procedure (FEDP)

The following procedure applies to the field sampling devise decontamination procedure for the Flyway Site. A trowel or appropriate sampling devise will be decontaminated before soil samples are collected. The following decontamination procedure is listed below.

1. Remove all gross contamination with a plastic brush.

2. Use clean water and a plastic brush to wash each piece of equipment.

3. Remove excess water present on the equipment by shaking.

4. Use a paper towel to dry each piece of equipment.

5. Wrap dried equipment in aluminum foil.

6. Once a week all soil sampling equipment will be cleaned using Alconox and clean water.

Spent gloves, and PPE must be properly disposed.

If disposable sampling devises are used, this decontamination procedure will not apply.